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Acurex Corp, Mountain View, Calif Aerotherm Div

Prepared for

Environmental Protection Agency, San Francisco, Calif Region IX

Mar 77

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STACK TEST RESULTS AT PHELPS-DODGE CORPORATION, AJO, ARIZONA

Volume I

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485 Clyde Avenue
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March 1977

AEROTHERM FINAL REPORT 76-231

Prepared for

EPA Task Officer — L. Bowerman

Enforcement Division
Environmental Protection Agency, Region IX
100 California Street
San Francisco, California 94111

Contract 68-01-3158
Task 12

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In July 1976 at the request of EPA, Region IX, Enforcement Division, the Aerotherm Division of the Acurex Corporation conducted a series of air pollutant mass emissions tests at the new Cornelia Branch copper ~~smelte~~ smelter of the Phelps Dodge Corporation located in Ajo, Arizona. The tests were performed simultaneously at three locations: 1)the main stack, 2)the reverberatory furnace duct and 3)the converter side sulfuric acid plant tail gas duct. Tests for particulate matter (Method 5), sulfur dioxide and sulfuric acid (Method 8) were conducted at each location using a combined sampling train. In addition tests for "condensable particulate matter" were conducted at the main stack and the reverberatory furnace duct using the combined Method 5 and 8 sampling train with an instack filter added.

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AEROTHERM FINAL REPORT 76-231

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✓ 1). The fifth sentence under the heading Particulate Matter on p. 8-11 should read: "Based on these figures, there is 90-percent confidence that the measured particulate emission rate at the outlet of the acid plant is between 5.9 lb/hr and 7.3 lb/hr."

✓ 2). The second sentence under the heading SO_2 on p. 8-19 should read: "Based on the above data, there is 90-percent confidence that the measured SO_2 emission rate is between 3.7×10^3 lb/hr and 6.5×10^3 lb/hr."

✓ 3). The second sentence under the heading Particulate Matter on p. 8-25 should read: "Based on this data, there is 90-percent confidence that the measured particulate emission rate is between 486.9 lb/hr and 559.7 lb/hr.

TABLE OF CONTENTS

Section		Page
1	INTRODUCTION	1-1
2	PREPARATION OF THE SAMPLING EQUIPMENT	2-1
	2.1 Sampling Nozzles	2-1
	2.2 Sampling Probes	2-1
	2.3 Probe and Oven Heaters	2-1
	2.4 Umbilical Cords	2-2
	2.5 Vacuum Pumps	2-2
	2.6 Glass Impingers	2-2
	2.7 Glass Fiber Filters	2-2
	2.8 Chemicals	2-2
	2.9 In-Stack Filters	2-3
	2.10 Unirail Modifications	2-3
	2.11 Special Sampling Equipment	2-3
3	CALIBRATION PROCEDURES	3-1
	3.1 Digital Temperature Indicator and Thermocouples	3-1
	3.2 Dry Gas Meter and Orifice Meter	3-1
	3.3 S-Type Pitot Tube Calibration	3-13
	3.4 Differential Pressure Transducer	3-31
	3.5 Magnehelic Gauges	3-31
	3.6 Isopropanol	3-36
	3.7 Mettler Analytical Balance	3-36
4	SAMPLING EQUIPMENT	4-1
	4.1 SO_3/H_2SO_4, SO_2, and Particulate Sampling Equipment	4-1
	4.1.1 Sampling Probes	4-2
	4.1.2 Heated Oven	4-2
	4.1.3 Cyclone	4-2
	4.1.4 Filter Holder	4-11
	4.1.5 Impinger Train	4-11
	4.1.6 Control Console	4-17
	4.2 Stack Gas Composition and Moisture Sampling Equipment	4-17
	4.3 Stack Gas Composition	4-17
5	SAMPLING PROCEDURES	5-1
	5.1 Preliminary Measurements	5-1
	5.1.1 Acid Plant Outlet Sampling Site	5-1
	5.1.2 Reverb Furnace Electrostatic Precipitator Outlet Sampling Site	5-8
	5.1.3 Main Smelter Stack	5-12
	5.2 Preliminary Calculations	5-12
	5.3 EPA Method 5 and 8 Field Test Procedures	5-17
	5.3.1 Preparation of the Sampling Trains	5-17
	5.3.2 Use of the Sampling Trains	5-21
6	SAMPLE RECOVERY AND HANDLING PROCEDURES	6-1
	6.1 EPA Methods 5 and 8 Sample Recovery Procedures	6-1
	6.2 EPA Methods 3 and 4 Sample Recovery Procedures	6-9
	6.3 EPA Methods 5 and 8 Sample Handling Procedures	6-10

TABLE OF CONTENTS (Concluded)

Section Page

7 PHYSICAL AND CHEMICAL ANALYSIS PROCEDURES 7-1

7.1 Weighting the Particulate Filters 7-1
7.2 Nozzle, Probe and Filter Holder Acetone Washes 7-2
7.3 SO_3/H_2SO_4 and SO_2 Samples 7-2

7.3.1 Titration Procedures for SO_3/H_2SO_4 Samples 7-5
7.3.2 Titration Procedures for SO_2 Samples 7-6

8 RESULTS AND CONCLUSIONS 8-1

8.1 Calculating Process Weights and Allowable Emission Rates 8-1

8.1.1 Process Weight and Allowable Emission Rate for the Acid Plant 8-1
8.1.2 Process Weight and Allowable Emission Rate for the Reverb Furnace . . . 8-4
8.1.3 Process Weight and Allowable Emission Rate for the Main Stack 8-6

8.2 Results of the Field Tests 8-6
8.3 Discussion of Field Test Results 8-6

8.3.1 Acid Plant Test Results 8-6
8.3.2 Reverb Furnace ESP Outlet Test Results 8-13
8.3.3 Main Stack Test Results 8-19

8.4 Conclusions 8-31

LIST OF TABLES

Table Page

3-1 Meter Calibration Data for Method 5 and 8 Control Module for Acid Plant 3-8

3-2 Meter Calibration Data for Method 5 and 8 Control Module for Reverb ESP Outlet . 3-9

3-3 Meter Calibration Data for Method 3 and 4 Control Module for Reverb ESP Outlet . 3-10

3-4 Meter Calibration Data for Method 3 and 4 Control Module for Main Stack 3-11

3-5 Meter Calibration Data for Method 5 and 8 Control Module for Main Stack 3-12

3-6 Pitot Tube Calibration Data for Method 5 and 8 Probe for Acid Plant 3-23

3-7 Pitot Tube Calibration Data for Method 3 Probe for Acid Plant 3-24

3-8 Pitot Tube Calibration Data for Method 5 and 8 Probe With In-Stack Filter for Reverb ESP Outlet 3-25

3-9 Pitot Tube Calibration Data for Method 5 and 8 Probe Without In-Stack Filter for Reverb ESP Outlet 3-26

3-10 Pitot Tube Calibration Data for Method 3 and 4 Probe for Reverb ESP Outlet . . . 3-27

3-11 Pitot Tube Calibration Data for Method 5 and 8 Probe With In-Stack Filter for Main Stack 3-28

3-12 Pitot Tube Calibration Data for Method 5 and 8 Probe Without In-Stack Filter for Main Stack 3-29

3-13 Pitot Tube Calibration Data for Method 3 and 4 Probe for Main Stack 3-30

8-1 Summary of Sampling Data 8-7

8-2 Summary of Particulate, SO_3/H_2SO_4 and SO_2 Emission Data 8-8

8-3 Summary of Sampling Times 8-9

8-4 Comparison of EPA Method 5 and In-Stack/Out-of-Stack Sampling Techniques 8-16

8-5 Valid Particulate Emission Rate Data 8-22

8-6 Gas Flow Contribution of Fugitive Gas Collection System 8-24

8-7 Rated Fan Capacities in Fugitive Gas Collection System[a] 8-26

8-8 Valid SO_3/H_2SO_4 Emission Rate Data 8-27

8-9 Valid SO_2 Emission Rate Data 8-28

Pages v and vi
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SECTION 1

INTRODUCTION

At the request of EPA Region IX, the Aerotherm Division of the Acurex Corporation undertook a series of stack tests at the Phelps-Dodge Corporation copper smelter in Ajo, Arizona. These tests had three main purposes: (1) to measure particulate, SO_3/H_2SO_4, and SO_2 emission rates with fugitive gas collection system operating; (2) to measure particulate, SO_3/H_2SO_4, and SO_2 emission rates with the fugitive gas collection system not operating; (3) to compare in-stack sampling methods with EPA Method 5.

Simultaneous stack tests were conducted on the tail gas from the acid plant, on the effluent at the outlet of the reverb furnace electrostatic precipitator, and on the effluent from the main stack during the period from July 15, 1976 to July 30, 1976. In addition, Aerotherm personnel, with the assistance of various representatives from EPA Region IX, recorded process weight data during the stack tests.

This report documents the preparations and procedures used by Aerotherm at Ajo, Arizona. Section 2 describes the equipment preparation made by Aerotherm for the stack tests. Section 3 describes the calibration procedures and Section 4 details the sampling equipment used. Sampling procedures are discussed in Section 5. Sample recovery and handling procedures are described in Section 6, and the chemical analysis procedures used to analyze the samples collected during the stack tests are described in Section 7. Finally, Section 8 presents the results of the stack tests.

Three appendices which contain summaries of the daily activities and the raw data collected during the various stages of the stack tests are also included in the report.

SECTION 2

PREPARATION OF THE SAMPLING EQUIPMENT

Before stack tests in the field can be conducted, sampling equipment must be carefully prepared to make reliable field measurements and to minimize costly delays due to equipment breakdown. This section of the report describes the preparations made by Aerotherm personnel prior to conducting the field tests in Ajo, Arizona.

2.1 SAMPLING NOZZLES

All sampling nozzles to be used in the field tests were inspected — and sharpened if necessary — to minimize both gas stream flow disturbances and particulate impacting on the edge of the nozzle. Those nozzles which had been nicked or damaged were sharpened. The internal diameters of the sampling nozzles were measured with a micrometer to insure accurate values for the nozzle diameter in calculating the isokinetic sampling rates for the particulate tests.

2.2 SAMPLING PROBES

At the request of EPA Region IX, Pyrex glass liners were inserted into all the particulate sampling probes. In addition, spare glass liners were purchased to replace any liner which might accidently break in the field. Stainless steel probe liners were used in all moisture sampling trains.

2.3 PROBE AND OVEN HEATERS

Before the equipment was packed, the probe and oven heaters and their temperature controllers were checked out in the laboratory by setting the temperature controllers for the probe heater and the oven heater at 250°F and turning the heaters on. The system was left on for approximately 5 minutes to make sure that the heaters were working and the temperature controllers were holding the temperatures at 250°F.

2.4 UMBILICAL CORDS

The umbilical cord which connects the probe to the control module was inspected and the polyflow tubing inside the umbilical cord checked for cracks. The quick disconnects were also inspected and checked to make sure they were operating properly.

2.5 VACUUM PUMPS

The vacuum pumps were operated in the laboratory to measure their freeflow capacity and leak rate at maximum vacuum. All vacuum pumps were found to be leak tight.

2.6 GLASS IMPINGERS

At the request of EPA Region IX, the impinger train for the particulate tests was made to comply with EPA Method 8. Spare impingers and bubblers were purchased to account for any breakage which might occur during the field tests, and an extra isopropanol impinger was added to the sampling train to counteract evaporation from the first bubbler. In addition, a blank impinger was inserted between the last hydrogen peroxide impinger and the silica gel impinger to prevent silica gel from backing up into the peroxide solution.

2.7 GLASS FIBER FILTERS

The glass fiber filters were obtained from Schleicher and Schuell and were equivalent to the MSA 1106 BH glass fiber filters specified by EPA Method 5. The filters were placed in an oven at 300°F for 3 hours, then placed into individual plastic petri dishes and put in a desiccator for conditioning over silica gel for at least 24 hours. The filters were then taken to the balance room and weighed to the nearest 0.01 milligram on a Mettler Model H20 analytical balance. After weighing, each filter was returned to the plastic petri dish, which was sealed with masking tape and labeled with the filter number and tare weight. This information was also recorded in the filter record book.

2.8 CHEMICALS

Several types of chemicals were purchased for the field tests. Analytical reagent grade acetone from Mallinckrodt was used to rinse the front half of the sampling train. Technical grade acetone from the McKesson Chemical Company was used to clean the impingers after the samples had been recovered from the impingers. Technical grade acetone was used for routine glassware cleaning instead of reagent grade acetone, since it was inexpensive and adequate for the task at hand. Pure isopropanol — also from Mallinckrodt — was mixed with distilled water from Phelps-Dodge Laboratory to make a fresh 80 percent IPA solution.

Analytical reagent grade H_2O_2 in 30 percent solution from Mallinckrodt was used to make fresh solutions of H_2O_2 each day. A 20 percent H_2O_2 solution was used to collect SO_2 in the stack gases from the reverb furnace electrostatic precipitator, and a 3 percent solution was used to collect SO_2 in the stack gases from the acid plant and main stack.

Silica gel grade Type 4, from MC/B Company, was used to dry the sample gases. Silica gel is equivalent to Drierite, the desiccant specified by EPA Method 5.

Chemicals used to analyze the samples collected in the field were deionized water from the Aerotherm Source Evaluation Laboratory; analytical reagent grade thorin indicator and sulfuric acid from Baker; barium perchlorate from Barium & Chemicals, Inc.; sodium hydroxide, a 1 normal Acculute standard volumetric solution, from Anachemica Chemicals Limited.

2.9 IN-STACK FILTERS

Special S-type pitot tube tips were fabricated to accommodate the stainless steel 47-mm filter holders. When this filter holder was attached to the sampling probe, the S-type pitot tube tips were adjacent to the sampling nozzle opening. The glass fiber filters used in the in-stack filter holders were obtained from Schleicher and Schuell and were equivalent to the MSA 1106 BH filters specified by EPA Method 5.

2.10 UNIRAIL MODIFICATIONS

To use the existing unirails at the main stack test site, adjustable brackets were fabricated to support the sampling probe and oven and keep them horizontal at all times.

2.11 SPECIAL SAMPLING EQUIPMENT

For these tests Aerotherm purchased (at no cost to the government) Validyne Engineering's Model DP103 pressure transducer with a full scale reading of 0.01 psi, a Model CD12 analog indicator, and a Model PN7110-15 cable to deliver the signal from the pressure transducer to the analog indicator. The transducer was used instead of the magnehelic gauges normally used to read the velocity pressure in the stack in combination with the S-type pitot tube. Magnehelic gauges are not suitable for use at the low velocity pressures that occur in the kind of tests performed for this project.

SECTION 3

CALIBRATION PROCEDURES

After the sampling equipment was inspected and prepared, various pieces of the sampling trains were calibrated. This section describes these calibration procedures.

3.1 DIGITAL TEMPERATURE INDICATOR AND THERMOCOUPLES

The Aerotherm High Volume Stack Sampler uses a digital temperature indicator with an 8-point thermocouple selector switch. The selector switch permits temperature to be monitored at each of the following locations: stack gas, probe, oven, impinger train outlet, gas meter inlet, gas meter outlet, and 2 spare locations. The digital temperature indicator operates over a range of 0°F to 1500°F with an accuracy of ±4°F. All temperatures in the sampling train are measured by chromel-alumel thermocouples.

The digital temperature indicator for each control module of each sampling train was checked in two ways. For the first check, a thermocouple was immersed in an ice bath at 32°F and the leads from the thermocouple connected to one of the spare locations on the control module. The reading on the digital temperature indicator was then observed and compared with that of the ice bath. For the second check, the oven controller was set at 250°F. The temperature of the oven was then displayed by the digital temperature indicator and compared to the temperature as measured by a Fluke Model 2100A digital thermometer calibrated twice a year against an NBS traceable standard. The zero and span of each digital temperature indicator was adjusted if the readings disagreed with either the ice bath or the Fluke digital thermometer readings.

3.2 DRY GAS METER AND ORIFICE METER

The control module of the sampling train contains a dry gas meter and an orifice meter. The dry gas meter measures the total volume of gas sampled during the test. The orifice meter is used to measure the rate at which the stack gas is sampled.

A Rockwell Model 415 gas meter (illustrated in Figure 3-1) is used to simultaneously calibrate the dry gas meter and orifice meter in each control module. This calibration standard is the same type of meter that is used in the control module of each sampling train. The accuracy of the

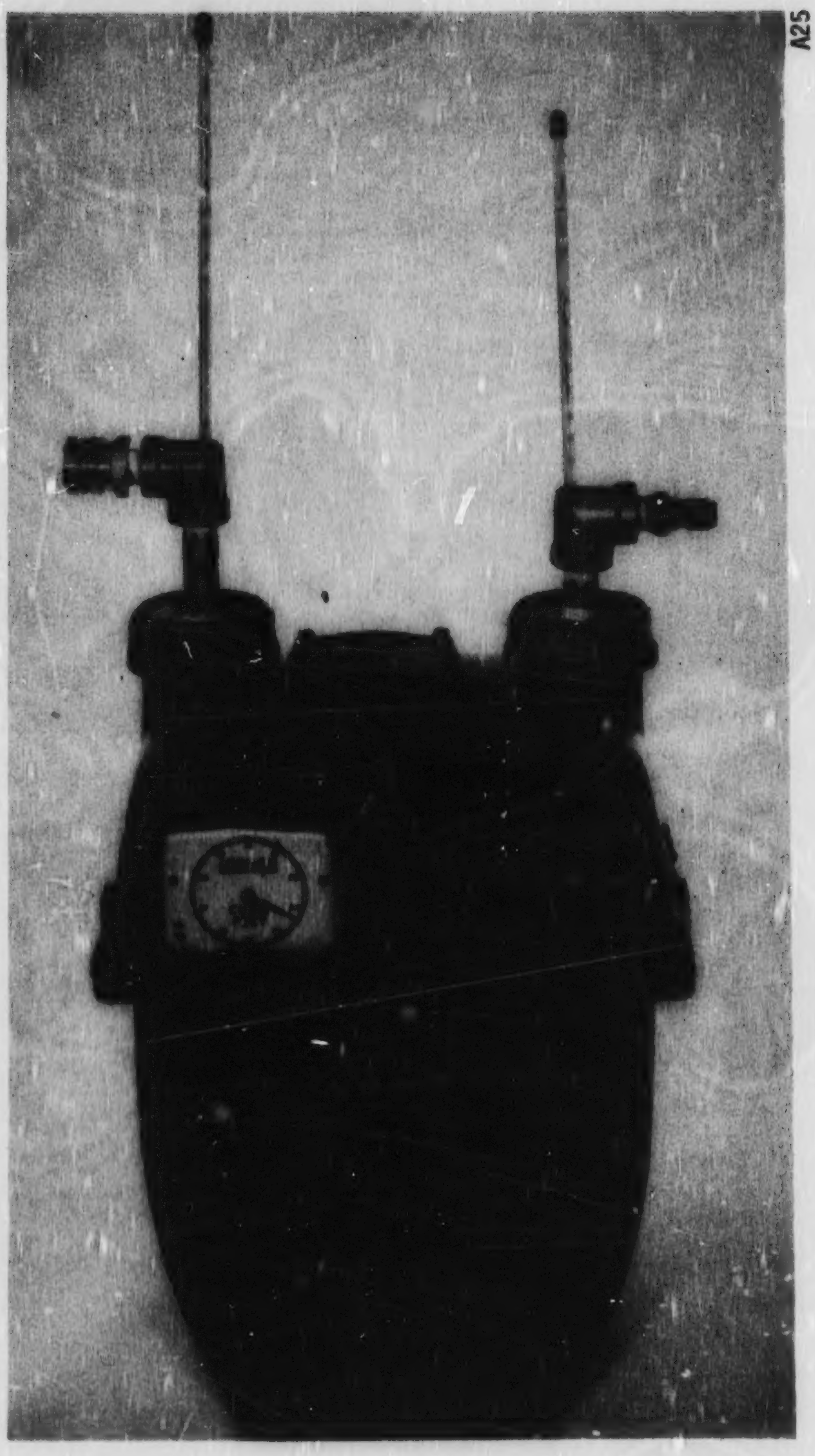
A25

Figure 3-1. Rockwell Model 415 gas meter calibration standard.

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Rockwell meter has been verified against a National Bureau of Standards traceable bell prover maintained by the Pacific Gas & Electric Company. In addition, the Rockwell meter never leaves Aerotherm's Source Evaluation Laboratory.

Before performing a calibration, the Rockwell gas meter and the control module to be calibrated were left overnight in the Source Evaluation Laboratory to come to thermal equilibrium. The next day, the magnehelic gauges in the control module were zeroed and leveled and the following components were arranged in series: a vacuum pump, the control module to be calibrated, and the calibration standard. Calibration information and the initial readings on the calibration standard gas meter and the gas meter in the control module were recorded on the data sheet shown in Figure 3-2.

The vacuum pump was then turned on with the valves closed. Next, the valves were opened, the ΔH across the orifice magnehelic gauge was set at a particular value, and the timer in the control module was started. The initial inlet and outlet temperatures at the calibration standard meter were measured with standard mercury-in-glass thermometers. The inlet and outlet temperatures of the dry gas meter in the control module were measured by thermocouples installed in the meter and the temperatures were displayed on the digital temperature indicator. The flowrate through the assembled components was maintained at a constant rate (as indicated by the reading on the orifice magnehelic gauge) for a known length of time, ranging from 5 to 10 minutes, until at least 5 cubic feet of air had passed through the system.

At the completion of a calibration run, the final dry gas meter readings and the final inlet and outlet dry gas meter temperatures were recorded. The same procedure was repeated at four different flowrates to calibrate the orifice meter over its entire useful range. The data from these calibrations were then used to calculate the calibration constants for the dry gas meter and the orifice meter in each control module.

The formula used to calculate the calibration constant for the dry gas meter is as follows:

$$\alpha = \frac{V_d \left(P_{bar} + \frac{\Delta H_o}{13.6}\right)\left(\frac{T_{pi} + T_{po}}{2} + 460\right)}{V_p P_{bar}\left(\frac{T_{di} + T_{do}}{2} + 460\right)}$$

where

α = calibration constant for dry gas meter in the control module

V_d = total volume of gas passing through the dry gas meter in the control module, ft³

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Date
Time
Barometric Pressure
Ambient Temperature

Orifice Meter
Orifice Magnehelic
Primary Calibration Meter
Control Module

Operators
Wet Bulb Temperature

METER CALIBRATION DATA

Orifice Manometer ΔH_{ref} (in.wg.)	Orifice Magnehelic ΔH_0 (in.wg.)	Primary Meter ΔH_p (in.wg.)	Dry Test Meter P_{dg} (in. wg.)	Gas Volume Primary Meter V_p (ft.³)	Gas Volume Dry Test Meter V_d (ft.³)	Temperature						Time t (min.)	α	K_0
						Primary Meter			Dry Test Meter					
						Inlet, T_{pi} (°F)	Outlet, T_{po} (°F)	Avg., T_{pa} (°F)	Inlet, T_{di} (°F)	Outlet, T_{do} (°F)	Avg., T_{da} (°F)			
	0.2													
	0.4													
	0.6													
	0.8													
	1.0													
	1.2													
	1.4													
	1.6													
	1.8													
	2.0													
	2.2													
	2.4													
	2.6													
	2.8													
	3.0													
	3.2													
	3.4													
	3.6													
	3.8													
	4.0													
	4.2													
	4.4													
	4.6													
	4.8													
	5.0													
													Average	

Figure 3-2. Meter calibration data sheet.

V_p = total volume of gas passing through the calibration standard meter, ft^3

P_b = barometric pressure, inches Hg

ΔH_o = pressure drop across the orifice meter in the control module, inches H_2O

13.6 = conversion factor, inches H_2O/inches Hg

T_{di} = inlet temperature of dry gas meter in the control module, °F

T_{do} = outlet temperature of dry gas meter in the control module, °F

T_{pi} = inlet temperature of calibration standard meter, °F

T_{po} = outlet temperature of calibration standard meter, °F

460 = conversion factor, °F to °R

The meter readings obtained during the field tests were divided by α to obtain the true volume.

The calibration constant for the orifice meter was determined using the following formula:

$$K_o = \frac{V_p}{t\left(\frac{T_{do}\,\Delta H_o}{P_{bar}\,M_m}\right)^{1/2}}$$

where

K_o = calibration constant for the orifice meter in the control module

V_p = total volume of gas passing through the calibration standard meter, ft^3

T_{do} = outlet temperature of dry gas meter in the control module, °R

t = calibration time, minutes

ΔH_o = pressure drop across the orifice meter in the control module, inches H_2O

P_{bar} = barometric pressure, inches Hg

M_m = molecular weight of the air, 28.96 lb/lb-mole

An average calibration constant was determined for the dry gas meter and orifice meter in each control module by averaging the individual test results. The calibration data for each control module used during the field tests is presented in Tables 3-1 to 3-5.

TABLE 3-1. METER CALIBRATION DATA FOR METHOD 5 AND 8 CONTROL MODULE FOR ACID PLANT

Date 7/6
Time 3:15 pm
Barometric Pressure 30
Ambient Temperature 73

Orifice Meter small orifice (.186)
Orifice Magnehelic 40725 OB 21
Primary Calibration Meter DGM 259453
Control Module 0.0009

Operators George Sutton, Ross Gilchrist
Wet Bulb Temperature

METER CALIBRATION DATA

Orifice Manometer ΔH_{ref} (in.wg.)	Orifice Magnehelic ΔH_D (in.wg.)	Primary Meter ΔH_p (in.wg.)	Dry Test Meter P_{dg} (in. wg.)	Gas Volume Primary Meter V_p (ft.³)	Gas Volume Dry Test Meter V_d (ft.³)	Temperature						Time t (min.)	γ	K_o
						Primary Meter			Dry Test Meter					
						Inlet, T_{pi} (°F)	Outlet, T_{po} (°F)	Avg., T_{pa} (°F)	Inlet, T_{di} (°F)	Outlet, T_{do} (°F)	Avg., T_{da} (°F)			
	0.2													
	0.4													
	0.6													
	0.8													
	1.0			5.400	5.419	75 75	75 75	75	79 78	79 77	78	10	1.000	0.687
	1.2													
	1.4													
	1.6													
	1.8													
2.0	2.0			6.048	6.051	75 75	75 75	75	77 77	76 76	76.5	8	1.003	0.682
	2.2													
	2.4													
	2.6													
	2.8													
	3.0			7.324	7.304	75 75	75 75	75	77 78	76 75	76.5	8	1.001	0.675
	3.2													
	3.4													
	3.6													
	3.8													
3.98	4.0			5.214	5.208	75 75	75 75	75	78 78	75 75	76.5	5	1.006	0.667
	4.2													
	4.4													
	4.6													
	4.8													
	5.0			5.884	5.840	75 75	75 75	75	79 79	76 75	77.5	5	1.001	0.673
													Average	
													1.002	0.677

T-57

TABLE 3-2. METER CALIBRATION DATA FOR METHOD 5 AND 8 CONTROL MODULE FOR REVERB ESP OUTLET

Date 7/6
Time 12:50 pm
Barometric Pressure 29.99
Ambient Temperature 73

Orifice Meter small orifice (0.186)
Orifice Magnehelic 40725 AM 7
Primary Calibration Meter DGM 259453
Control Module 0.0015

Operators George Sutton, Ross Gilchrist
Wet Bulb Temperature

METER CALIBRATION DATA

T-68

Orifice Manometer ΔH_{ref} (in.wg.)	Orifice Magnehelic ΔH_D (in.wg.)	Primary Meter ΔH_p (in.wg.)	Dry Test Meter P_{dg} (in. wg.)	Gas Volume Primary Meter V_p (ft.³)	Gas Volume Dry Test Meter V_d (ft.³)	Temperature						Time t (min.)	γ	K_{θ}
						Primary Meter			Dry Test Meter					
						Inlet, T_{pi} (°F)	Outlet, T_{po} (°F)	Avg., T_{pa} (°F)	Inlet, T_{di} (°F)	Outlet, T_{do} (°F)	Avg., T_{da} (°F)			
	0.2													
	0.4													
	0.6													
	0.8													
	1.0			5.363	5.415	73 73	73 73	73	77 76	76 74	75.75	10	1.007	0.684
	1.2													
	1.4													
	1.6													
	1.8													
1.98	2.0			5.983	6.025	74 74	73 74	74	76 76	74 73	74.75	8	1.010	0.676
	2.2													
	2.4													
	2.6													
	2.8													
	3.0			7.326	7.347	75 75	74 74	74.5	76 78	73 73	75.0	8	1.009	0.677
	3.2													
	3.4													
	3.6													
	3.8													
3.98	4.0			5.253	5.276	75 75	74 74	74.5	77 79	73 72	75.25	5	1.013	0.674
	4.2													
	4.4													
	4.6													
	4.8													
	5.0			5.975	5.964	75 76	74 74	75	79 81	72 73	76.25	5	1.009	0.687
													Average	
													1.010	0.680

TABLE 3-3. METER CALIBRATION DATA FOR METHOD 3 AND 4 CONTROL MODULE FOR REVERB ESP OUTLET

Date 7/7	Orifice Meter small orifice (0.187)	Operators George Sutton, Ross Gilchrist
Time 8:25 am	Orifice Magnehelic 41112 NH 18	Wet Bulb Temperature
Barometric Pressure 30	Primary Calibration Meter DGM 259453	
Ambient Temperature 72	Control Module 0.0017	

METER CALIBRATION DATA

Orifice Manometer ΔH_{ref} (in.wg.)	Orifice Magnehelic ΔH_O (in.wg.)	Primary Meter ΔH_p (in.wg.)	Dry Test Meter P_{dg} (in. wg.)	Gas Volume Primary Meter V_p (ft.³)	Gas Volume Dry Test Meter V_d (ft.³)	Temperature, Primary Meter, Inlet, T_{pi} (°F)	Temperature, Primary Meter, Outlet, T_{po} (°F)	Temperature, Primary Meter, Avg., T_{pa} (°F)	Temperature, Dry Test Meter, Inlet, T_{di} (°F)	Temperature, Dry Test Meter, Outlet, T_{do} (°F)	Temperature, Dry Test Meter, Avg., T_{da} (°F)	Time t (min.)	γ	K_0
	0.2													
	0.4													
	0.6													
	0.8													
1.08	1.0			5.405	5.418	73 72	72 72	72	64 64	64 64	64	10	1.020	0.697
	1.2													
1.48	1.4													
	1.6													
	1.8													
2.12	2.0			6.044	6.041	72 72	72 72	72	64 65	64 64	64	8	1.019	0.690
	2.2													
2.41	2.4													
	2.6													
	2.8													
3.10	3.0			7.274	7.271	72 72	72 72	72	65 67	64 65	65	8	1.020	0.678
	3.2													
3.50	3.4													
	3.6													
3.91	3.8													
	4.0			5.203	5.174	72 72	72 72	72	67 68	65 65	66	5	1.015	0.673
	4.2													
	4.4													
	4.6													
	4.8													
	5.0			5.770	5.770	72 72	72 72	72	69 70	65 65	67	5	1.021	0.668
													Average	
													1.018	0.681

TABLE 3-4. METER CALIBRATION DATA FOR METHOD 3 AND 4
CONTROL MODULE FOR MAIN STACK

Date 7/6
Time 11:00 am
Barometric Pressure 29.99
Ambient Temperature 72

Orifice Meter Small orifice (0.187)
Orifice Magnehelic 50523 MH63
Primary Calibration Meter DGM 259453
Control Module 0.0038

Operators George Sutton, Ross Gilchrist
Wet Bulb Temperature

METER CALIBRATION DATA

T-70

Orifice Manometer ΔH_{ref} (in.wg.)	Orifice Magnehelic ΔH_0 (in.wg.)	Primary Meter ΔH_p (in.wg.)	Dry Test Meter P_{dg} (in. wg.)	Gas Volume Primary Meter V_p (ft.³)	Gas Volume Dry Test Meter V_d (ft.³)	Temperature						Time t (min.)	Y	K_o
						Primary Meter			Dry Test Meter					
						Inlet, T_{pi} (°F)	Outlet, T_{po} (°F)	Avg., T_{pa} (°F)	Inlet, T_{di} (°F)	Outlet, T_{do} (°F)	Avg., T_{da} (°F)			
	0.2													
	0.4													
	0.6													
	0.8													
1.14	1.0			5.968	6.188	74 74	74 74	74	73 76	73 74	74	10	1.039	0.762
	1.2													
1.53	1.4													
	1.6													
	1.8													
2.12	2.0			6.443	6.660	74 75	74 74	74.25	77 80	74 76	76.75	8	1.033	0.727
	2.2													
2.52	2.4													
	2.6													
	2.8													
3.10	3.0			7.691	7.945	75 75	74 74	74.5	81 84	76 77	79.5	8	1.031	0.709
	3.2													
3.52	3.4													
	3.6													
3.94	3.8													
	4.0			5.523	5.698	75 76	74 74	74.75	84 86	77 77	81	5	1.030	0.706
	4.2													
	4.4													
	4.6													
	4.8													
	5.0			6.168	6.339	76 76	74 75	75.25	86 88	78 78	82.5	5	1.027	0.705
													Average	
													1.032	.722

TABLE 3-5. METER CALIBRATION DATA FOR METHOD 5 AND 8 CONTROL MODULE FOR MAIN STACK

Date 7/6
Time 10:00 am
Barometric Pressure 29.99
Ambient Temperature 72

Orifice Meter small orifice (0.187)
Orifice Magnehelic 50523 PM 50
Primary Calibration Meter DGM 259453
Control Module 0.0039

Operators George Sutton, Ross Gilchrist
Wet Bulb Temperature

METER CALIBRATION DATA

T-71

Orifice Manometer ΔH_{ref} (in.wg.)	Orifice Magnehelic $\Delta H_@$ (in.wg)	Primary Meter ΔH_p (in.wg.)	Dry Test Meter P_{dg} (in. wg.)	Gas Volume Primary Meter V_p (ft.³)	Gas Volume Dry Test Meter V_d (ft.³)	Temperature						Time t (min.)	α	K_o
						Primary Meter			Dry Test Meter					
						Inlet, T_{pi} (°F)	Outlet, T_{po} (°F)	Avg., T_{pa} (°F)	Inlet, T_{di} (°F)	Outlet, T_{do} (°F)	Avg., T_{da} (°F)			
	0.2													
	0.4													
	0.6													
	0.8													
	1.0			5.442	5.503	72 73	72 72	72.25	70 72	70 71	70.75	10	1.015	0.697
	1.2													
	1.4													
	1.6													
	1.8													
1.98	2.0			6.169	6.191	73 73	72 72	72.5	72 74	71 71	72	8	1.009	0.699
	2.2													
	2.4													
	2.6													
	2.8													
	3.0			7.485	7.502	74 74	73 73	73.5	74 77	71 72	73.5	8	1.008	0.693
	3.2													
	3.4													
	3.6													
	3.8													
3.98	4.0			5.415	5.423	74 74	73 74	73.75	77 80	72 73	75.5	5	1.008	0.695
	4.2													
	4.4													
	4.6													
	4.8													
	5.0			6.089	6.073	74 75	73 74	74	80 83	72 73	77	5	0.995	0.700
													Average	
													1.007	0.697

3.3 S-TYPE PITOT TUBE CALIBRATION

The S-type pitot tube attached to the sampling probe measures the volumetric stack gas flowrate and the velocity at each sampling point so that an isokinetic sampling rate can be determined. Before being taken into the field, all S-type tubes for each sampling train were calibrated in the Aerotherm Wind Tunnel illustrated in Figure 3-3, using the United Sensor Hemispherical nose standard pitot tube shown in Figure 3-4. The coefficient for this standard pitot tube was 0.99.

The velocity pressure sensed by the standard pitot tube was indicated on the Dwyer inclined manometer shown in Figure 3-5. This manometer has a full scale range of 0 to 4 inches of water and can measure differences as small as 0.01 inches of water. In addition, the inclined manometer is equipped with a valve system which allows the operator to change from the standard pitot tube to the S-type tube being calibrated without disconnecting any tubes, thereby eliminating any unnecessary movement of the manometer. The valve system on the manometer also has a vent position which allows the manometer to be zeroed at any time. The manometer has a built-in level gauge, so that the operator can check the level at any time.

The following steps were taken in calibrating S-type pitot tubes. First, the wind tunnel was leveled to insure that the test section was horizontal. The wind tunnel fan was then turned on and the outlet dampers were adjusted to provide the desired velocity in the wind tunnel. The inclined manometer was positioned on a box near the wind tunnel and the standard pitot tube was connected to the inclined manometer with tygon tubing. At the same time, the S-type pitot tube was connected to the manometer using polyflow tubing and quick disconnects. Both the tygon and polyflow lines were inspected to make sure there were no leaks or cracks. The inclined manometer was then zeroed and leveled. Before beginning the actual calibration run, the data at the top of the pitot tube calibration data sheet, Figure 3-6, was recorded.

The standard pitot tube was inserted into the wind tunnel through holes on the far side of the tunnel, as shown in Figure 3-3, with the nose of the pitot tube positioned at the centerline of the wind tunnel facing directly into the gas stream lines. The operator could look through a plexiglass window to insure that the pitot tube was pointed in the proper direction at all times. With the standard pitot tube properly positioned in the test section, the valve system on the inclined manometer was opened. The velocity pressure measured by the standard pitot tube and indicated on the inclined manometer was recorded and the standard pitot tube was withdrawn from the wind tunnel.

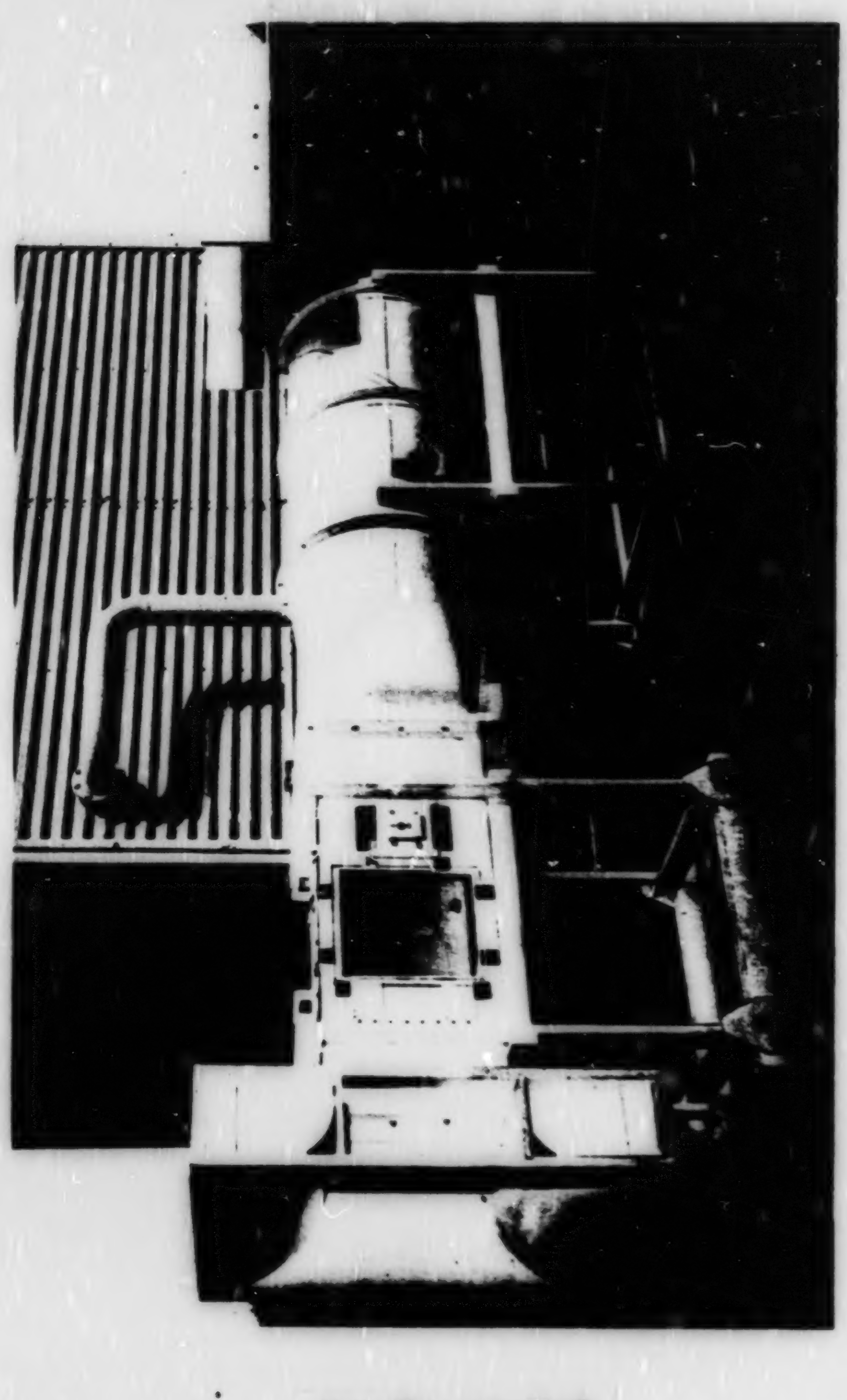

Figure 3-3. Aerotherm calibration wind tunnel.

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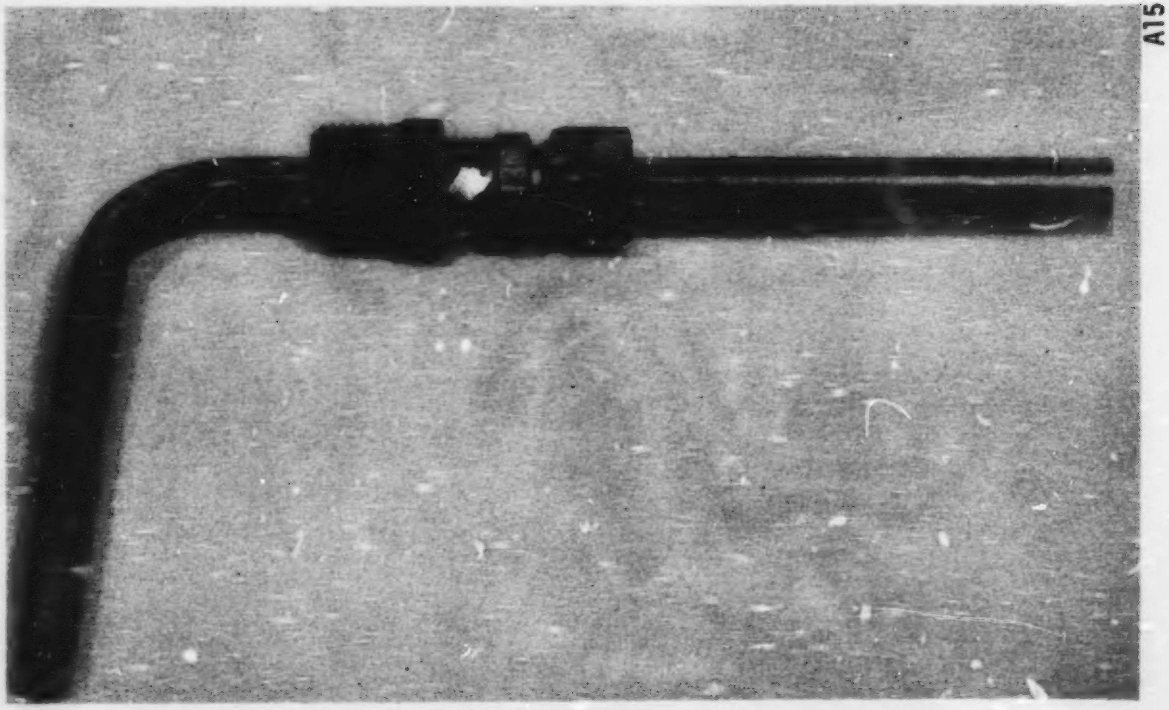

Figure 3-4. United Sensor hemispherical nose standard pitot tube.

A22

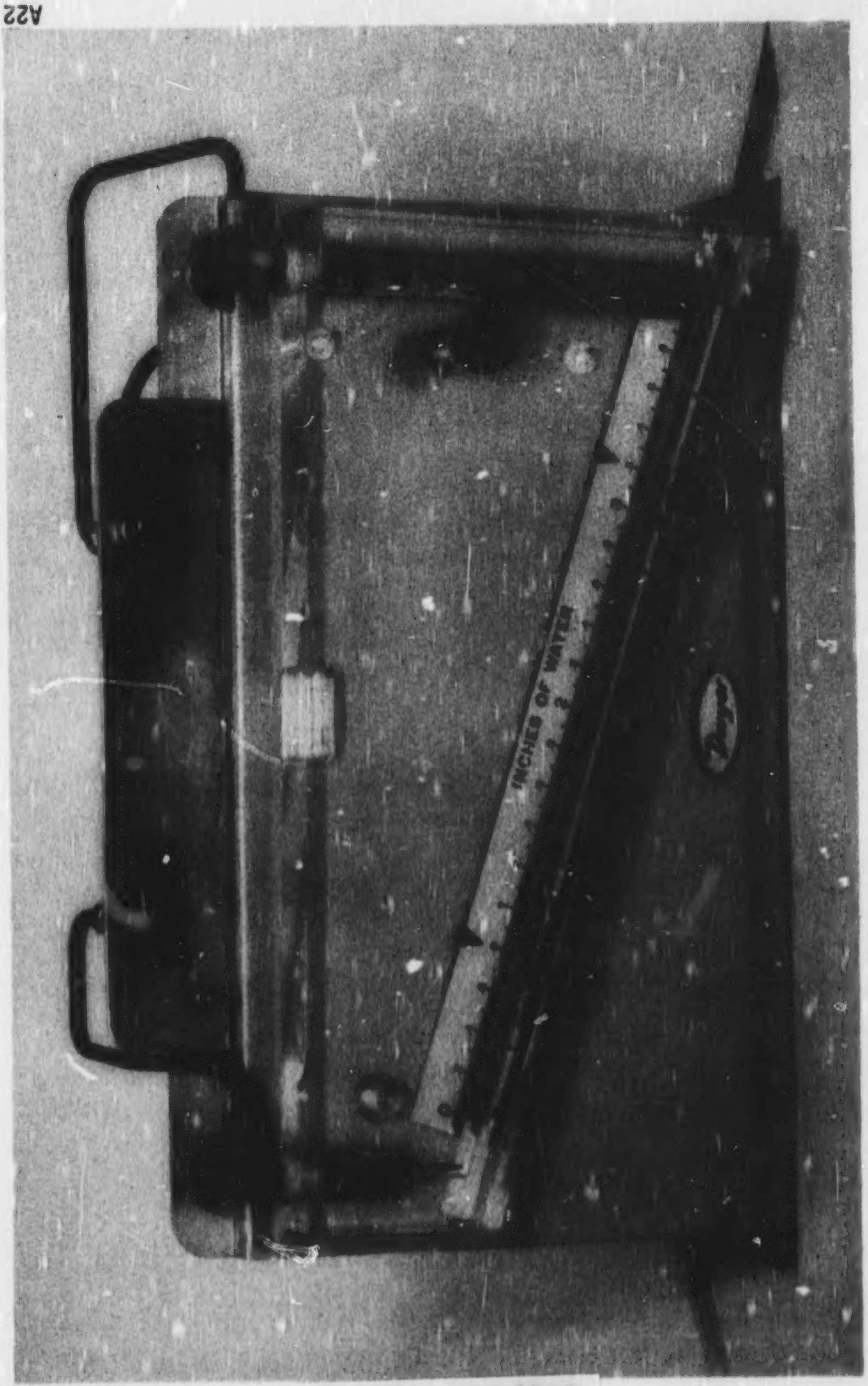
INCHES OF WATER
Dwyer

Figure 3-5. Dwyer inclined manometer.

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DATE
TIME
BAROMETRIC PRESSURE
AMBIENT TEMP

TEST PITOT TUBE
STANDARD PITOT TUBE
TEST SECTION LOCATION
OPERATORS

NOZZLE SIZE
NOZZLE-PITOT SPACING
PITOT-TC SPACING

PITOT TUBE CALIBRATION DATA

Test Pitot Tube ΔP (in. wg.)		Standard Pitot Tube ΔP (in. wg.)	C_p	Static Pressure (in. wg.)	Gas Temp (°F)	Wet Bulb Temp (F°)	Test Section Velocity (fps)
Upper Leg	Lower Leg						

Average $C_{p,\text{ upper}}$ =

Average $C_{p,\text{ lower}}$ =

Figure 3-6. Pitot tube calibration data sheet.

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The S-type pitot tube, attached to the sampling probe with the sampling nozzle and thermocouple in place, was then inserted through the near side of the wind tunnel so that the S-type tube tip was at the centerline of the wind tunnel. Inserting the sampling probe assembly into the wind tunnel was made easier by a bracket adjacent to the plexiglass window, as shown in Figure 3-3. The bracket was clamped tightly around the sampling probe assembly to insure that the assembly did not move in the wind tunnel. The bracket also made a seal with the inside of the wind tunnel wall so that the surfaces were flush and no flow disturbance was created at the wall. The tips of the S-type pitot tube were aligned and pointed directly into the gas stream lines, with the operator observing the position of the tips through the plexiglass window.

The valve system on the inclined manometer was then switched to the S-type pitot tube, and the velocity pressure measured by the lower leg of the S-type pitot tube and indicated by the inclined manometer was recorded. The sampling probe and pitot tube were then rotated 180 degrees, and the position of S-type pitot tube tips was rechecked. The velocity pressure measured by the upper leg of the S-type pitot tube was then recorded on the data sheet. This procedure was repeated several times in order to calibrate the S-type pitot tube over a wide range of velocities.

Separate calibrations of the S-type pitot tubes were also made with the in-stack filter attached to the sampling probe.

The formula used to calculate the coefficient for the S-type pitot tube for each sampling probe is as follows:

$$C_{P_{S\text{-type}}} = 0.99 \left(\frac{\Delta P_{std}}{\Delta P_{S\text{-type}}} \right)^{1/2}$$

where

$C_{P_{S\text{-type}}}$ = coefficient of the S-type pitot tube

ΔP_{std} = velocity pressure measured by the standard pitot tube, inches H_2O

$\Delta P_{S\text{-type}}$ = velocity pressure measured by the S-type pitot tube, inches H_2O

0.99 = coefficient of the standard pitot tube

The average coefficients for the upper and lower legs of the S-type pitot tube were calculated by averaging the individual coefficients of each calibration run. These average values were recorded on the calibration data sheet. Tables 3-6 to 3-13 summarize all the calibration data for the S-type pitot tubes and sampling probes that were used during the field test program.

TABLE 3-6. PITOT TUBE CALIBRATION DATA FOR METHOD 5 AND 8 PROBE FOR ACID PLANT

DATE 7-9-76
TIME 7:20 am
BAROMETRIC PRESSURE 30.00" Hg
AMBIENT TEMP

TEST PITOT TUBE 5' Acid Plant 5/8
STANDARD PITOT TUBE United Sensor
TEST SECTION LOCATION Center
OPERATORS George Sutton

NOZZLE SIZE 1/4"
NOZZLE-PITOT SPACING 1 1/32"
PITOT-TC SPACING 1"

PITOT TUBE CALIBRATION DATA

T-72

Test Pitot Tube ΔP (in. wg.)		Standard Pitot Tube ΔP (in. wg.)	C_p	Static Pressure (in. wg.)	Gas Temp (°F)	Wet Bulb Temp (F°)	Test Section Velocity (fps)
Upper Leg	Lower Leg						
0.34	0.34	0.20	0.759 0.759				
0.47	0.46	0.28	0.764 0.772				
0.57	0.56	0.36	0.787 0.794				
0.69	0.69	0.41	0.763 0.763				
0.85	0.84	0.52	0.774 0.779				
1.08	1.06	0.64	0.762 0.769				
1.19	1.18	0.77	0.796 0.800				
1.35	1.34	0.80	0.762 0.765				
1.53	1.49	0.90	0.759 0.769				
1.64	1.60	0.98	0.765 0.775				
1.8	1.78	1.07	0.763 0.768				
1.98	1.96	1.19	0.767 0.771				

Average $C_{p,\ upper}$ = 0.768

Average $C_{p,\ lower}$ = 0.774

TABLE 3-7. PITOT TUBE CALIBRATION DATA FOR METHOD 3 PROBE FOR ACID PLANT

DATE 7-12-76
TIME 10:40 am
BAROMETRIC PRESSURE 29.90" Hg
AMBIENT TEMP 76

TEST PITOT TUBE Special Acid Plant
STANDARD PITOT TUBE United Sensor
TEST SECTION LOCATION Center
OPERATORS George Sutton

NOZZLE SIZE
NOZZLE-PITOT SPACING
PITOT-TC SPACING

PITOT TUBE CALIBRATION DATA

T-73

Test Pitot Tube ΔP (in. wg.)		Standard Pitot Tube ΔP (in. wg.)	C_p	Static Pressure (in. wg.)	Gas Temp (°F)	Wet Bulb Temp (F°)	Test Section Velocity (fps)
Upper Leg	Lower Leg						
0.84	0.90	0.48	0.748 0.723				
1.08	1.06	0.61	0.744 0.751				
1.30	1.30	0.69	0.721 0.721				
1.54	1.58	0.90	0.757 0.747				
1.72	1.76	0.99	0.751 0.742				
2.12	2.19	1.20	0.745 0.734				

Average $C_{p,\ upper}$ = 0.744

Average $C_{p,\ lower}$ = 0.736

TABLE 3-8. PITOT TUBE CALIBRATION DATA FOR METHOD 5 AND 8 PROBE WITH IN-STACK FILTER FOR REVERB ESP OUTLET

DATE 7-9-76
TIME 10:20 am
BAROMETRIC PRESSURE 29.90" Hg
AMBIENT TEMP

TEST PITOT TUBE 10' Reverb w/filter
STANDARD PITOT TUBE United Sensor
TEST SECTION LOCATION Center
OPERATORS George Sutton

NOZZLE SIZE 7/16"
NOZZLE-PITOT SPACING 1-7/8"
PITOT-TC SPACING 15/16"

PITOT TUBE CALIBRATION DATA

T-74

Test Pitot Tube ΔP (in. wg.)		Standard Pitot Tube ΔP (in. wg.)	C_p	Static Pressure (in. wg.)	Gas Temp (°F)	Wet Bulb Temp (F°)	Test Section Velocity (fps)
Upper Leg	Lower Leg						
0.44	0.45	0.30	0.817 0.808				
0.56	0.54	0.37	0.805 0.819				
0.72	0.70	0.47	0.816 0.811				
0.5	0.5	0.33	0.804 0.804				
0.66	0.65	0.44	0.808 0.814				
0.78	0.8	0.52	0.808 0.798				
0.94	0.93	0.63	0.810 0.815				
1.08	1.06	0.70	0.797 0.804				
1.32	1.29	0.89	0.813 0.822				
1.20	1.18	0.79	0.803 0.810				

Average $C_{p,\ upper}$ = 0.808

Average $C_{p,\ lower}$ = 0.811

TABLE 3-9. PITOT TUBE CALIBRATION DATA FOR METHOD 5 AND 8 PROBE WITHOUT IN-STACK FILTER FOR REVERB ESP OUTLET

DATE 7-12-76
TIME 9:25 am
BAROMETRIC PRESSURE 29.99" Hg
AMBIENT TEMP 74

TEST PITOT TUBE 10' Reverb w/o filter
STANDARD PITOT TUBE United Sensor
TEST SECTION LOCATION Center
OPERATORS George Sutton

NOZZLE SIZE 7/16"
NOZZLE-PITOT SPACING 25/32"
PITOT-TC SPACING 15/16"

PITOT TUBE CALIBRATION DATA

T-75

Test Pitot Tube ΔP (in. wg.)		Standard Pitot Tube ΔP (in. wg.)	C_p	Static Pressure (in. wg.)	Gas Temp (°F)	Wet Bulb Temp (F°)	Test Section Velocity (fps)
Upper Leg	Lower Leg						
0.50	0.50	0.31	0.779 0.779				
0.58	0.60	0.37	0.791 0.777				
0.70	0.70	0.44	0.785 0.785				
0.79	0.78	0.51	0.795 0.800				
0.95	0.95	0.60	0.787 0.787				
1.1	1.08	0.68	0.778 0.785				
1.28	1.30	0.80	0.783 0.777				
1.50	1.52	0.94	0.784 0.778				

Average $C_{p,\ upper}$ = 0.785

Average $C_{p,\ lower}$ = 0.784

TABLE 3-10. PITOT TUBE CALIBRATION DATA FOR METHOD 3 AND 4 PROBE FOR REVERB ESP OUTLET

DATE 7-8-76
TIME 9:00 am
BAROMETRIC PRESSURE 30.03" Hg
AMBIENT TEMP

TEST PITOT TUBE 5' Reverb 3/4
STANDARD PITOT TUBE United Sensor
TEST SECTION LOCATION Center
OPERATORS George Sutton

NOZZLE SIZE 1/4"
NOZZLE-PITOT SPACING 15/16"
PITOT-TC SPACING 1-3/16"

PITOT TUBE CALIBRATION DATA

T-76

Test Pitot Tube ΔP (in. wg.)		Standard Pitot Tube ΔP (in. wg.)	C_p	Static Pressure (in. wg.)	Gas Temp (°F)	Wet Bulb Temp (F°)	Test Section Velocity (fps)
Upper Leg	Lower Leg						
0.42	0.42	0.26	0.779 0.779				
0.58	0.56	0.36	0.780 0.794				
0.68	0.72	0.44	0.796 0.774				
0.82	0.80	0.50	0.773 0.783				
1.02	1.06	0.62	0.772 0.757				
1.12	1.14	0.68	0.771 0.765				
1.30	1.28	0.78	0.767 0.773				
1.40	1.40	0.86	0.776 0.776				
1.50	1.48	0.92	0.775 0.781				

Average $C_{p,\ upper}$ = 0.777

Average $C_{p,\ lower}$ = 0.776

TABLE 3-11. PITOT TUBE CALIBRATION DATA FOR METHOD 5 AND 8 PROBE WITH IN-STACK FILTER FOR MAIN STACK

DATE 7-13-76
TIME 9:40 am
BAROMETRIC PRESSURE
AMBIENT TEMP

TEST PITOT TUBE 10' Main stack w/filter
STANDARD PITOT TUBE United Sensor
TEST SECTION LOCATION Center
OPERATORS C. Stanley

NOZZLE SIZE 7/16"
NOZZLE-PITOT SPACING 1-5/16"
PITOT-TC SPACING 1"

PITOT TUBE CALIBRATION DATA

T-77

Test Pitot Tube ΔP (in. wg.)		Standard Pitot Tube ΔP (in. wg.)	C_p	Static Pressure (in. wg.)	Gas Temp (°F)	Wet Bulb Temp (F°)	Test Section Velocity (fps)
Upper Leg	Lower Leg						
0.163	0.166	0.108	0.805 0.798				
0.152	0.149	0.094	0.778 0.786				
0.116	0.113	0.069	0.764 0.773				
0.083	0.083	0.055	0.808 0.808				
0.0664	0.0554	0.0387	0.756 0.827				
0.0387	0.0304	0.0221	0.748 0.844				
0.0221	0.0166	0.0110	0.698 0.805				
EXPANDED SCALE (0 to 0.008)							
0.0190	0.019	0.012	0.786 0.786				
0.0124	0.0124	0.0078	0.785 0.785				
0.0066	0.0066	0.0045	0.817 0.817				
0.0045	0.0049	0.0029	0.794 0.761				

Average $C_{p,\ upper}$ = 0.765
0.795 (expanded)

Average $C_{p,\ lower}$ = 0.805
0.787 (expanded)

TABLE 3-12. PITOT TUBE CALIBRATION DATA FOR METHOD 5 AND 8 PROBE WITHOUT IN-STACK FILTER FOR MAIN STACK

DATE 7-13-76
TIME 10:20 am
BAROMETRIC PRESSURE
AMBIENT TEMP

TEST PITOT TUBE 10' Main stack w/o filter
STANDARD PITOT TUBE United Sensor
TEST SECTION LOCATION Center
OPERATORS C. Stanley

NOZZLE SIZE 7/16"
NOZZLE-PITOT SPACING 1-3/16"
PITOT-TC SPACING 1-1/8"

PITOT TUBE CALIBRATION DATA

T-78

Test Pitot Tube ΔP (in. wg.)		Standard Pitot Tube ΔP (in. wg.)	C_p	Static Pressure (in. wg.)	Gas Temp (°F)	Wet Bulb Temp (F°)	Test Section Velocity (fps)
Upper Leg	Lower Leg						
0.166	0.166	0.099	0.767 0.767				
0.130	0.130	0.080	0.778 0.778				
0.110	0.110	0.066	0.767 0.767				
0.077	0.074	0.049	0.794 0.808				
0.0554	0.0554	0.0332	0.766 0.766				
0.0387	0.0387	0.0221	0.748 0.748				
0.0221	0.0221	0.0110	0.698 0.698				
EXPANDED SCALE (0 to 0.008)							
0.0149	0.0149	0.0091	0.773 0.773				
0.0091	0.0091	0.0049	0.726 0.726				
0.0045	0.0045	0.0024	0.722 0.722				

Average $C_{p,\ upper}$ = 0.759
0.751 (expanded)

Average $C_{p,\ lower}$ = 0.761
0.751 (expanded)

TABLE 3-13. PITOT TUBE CALIBRATION DATA FOR METHOD 3 AND 4 PROBE FOR MAIN STACK

DATE 7/12/76
TIME 8:30 am
BAROMETRIC PRESSURE 29.99" Hg
AMBIENT TEMP 70°F

TEST PITOT TUBE 10' Main Stack 3/4
STANDARD PITOT TUBE United Sensor
TEST SECTION LOCATION Center
OPERATORS George Sutton

NOZZLE SIZE 7/16"
NOZZLE-PITOT SPACING 15/16"
PITOT-TC SPACING 1-3/16"

T-79

PITOT TUBE CALIBRATION DATA

Test Pitot Tube ΔP (in. wg.)		Standard Pitot Tube ΔP (in. wg.)	C_p	Static Pressure (in. wg.)	Gas Temp (°F)	Wet Bulb Temp (F°)	Test Section Velocity (fps)
Upper Leg	Lower Leg						
0.04	0.04	0.02	0.700 0.700				
0.05	0.05	0.03	0.767 0.767				
0.09	0.09	0.05	0.758 0.738				
0.11	0.12	0.08	0.844 0.808				
0.13	0.12	0.08	0.777 0.808				
0.15	0.15	0.10	0.808 0.808				
0.24	0.24	0.14	0.756 0.756				
0.32	0.32	0.20	0.783 0.783				

Average $C_{p,\ upper}$ = 0.772

Average $C_{p,\ lower}$ = 0.771

Since it was known that the main stack at the Ajo smelter had a very low stack gas velocity, special calibration runs were performed on the particulate sampling probe and S-type pitot tube to be used on that stack. The calibration procedure was basically the same as that described above, except that the inclined manometer was replaced by a Validyne Model DP103 differential pressure transducer. A Validyne Model CD12 analog indicator was used to display the electrical signal from the differential pressure transducer. This pressure transducer and the analog indicator are illustrated in Figure 3-7. The transducer had three operating ranges: 0 to 0.2767 inches of water, 0 to 0.008 inches of water, and 0 to 0.0002 inches of water. As before, the pitot tube was calibrated over a range of velocities in the wind tunnel. All velocity pressures were less than 0.1 inches of water, since the velocity pressures at the main stack at the Ajo smelter were expected to be in this range.

3.4 DIFFERENTIAL PRESSURE TRANSDUCER

As mentioned in the previous section, Aerotherm purchased a differential pressure transducer from Validyne Engineering Corporation to measure low velocity pressures. At our request, Validyne calibrated the differential pressure transducer-indicator as a unit against a National Bureau of Standards traceable standard. This standard was an inclined manometer manufactured by the Meriam Instrument Company (Model No. 40 HE35) maintained in the Validyne Laboratory. Table 3-14 summarizes this calibration data.

3.5 MAGNEHELIC GAUGES

Normally, magnehelic gauges are used in the control module to display the velocity pressure in the stack and the pressure drop across the orifice meter. These magnehelic gauges were calibrated against the Dwyer inclined manometer described in Section 3.3.

The calibration procedure was a simple one. First, the Dwyer inclined manometer and the magnehelic gauge to be calibrated were zeroed and leveled in the control module. Tee taps were installed in both the high pressure and low pressure sides of the magnehelic gauge. One side of the Tee tap was connected to the orifice meter and the other side connected to the inclined manometer. A constant volume of gas was pumped through the orifice meter in the control module, and the readings on the magnehelic gauge and the Dwyer inclined manometer were observed. If the readings differed, the magnehelic gauge was adjusted and the calibration was repeated until the readings agreed. The calibration procedure was repeated at several different flowrates.

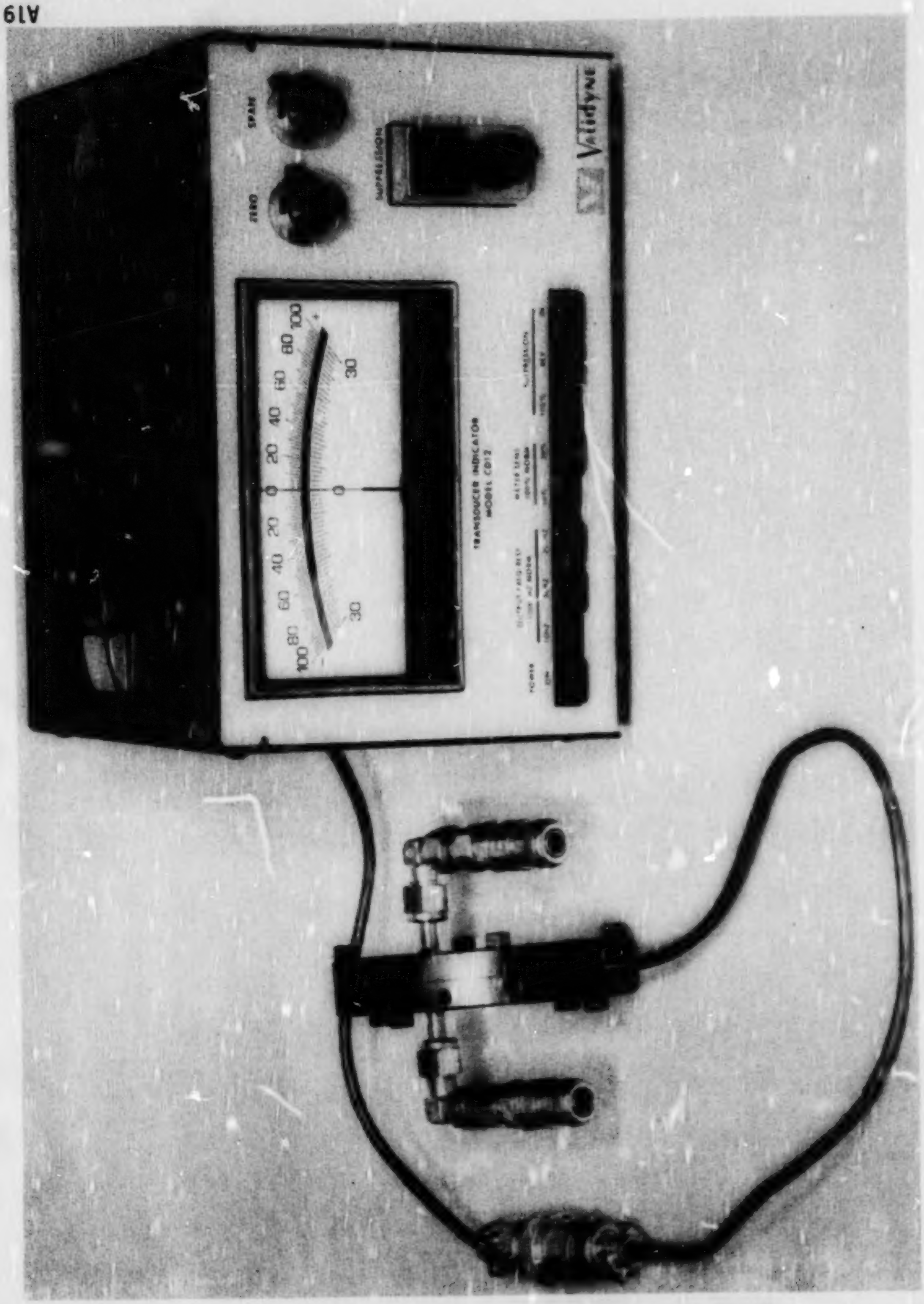
A19
Validyne
ZERO
SPAN
SUPPRESSION
TRANSDUCER INDICATOR
MODEL CD12
100
80
60
40
20
0
20
40
60
80
100
30
0
30
POWER
FILTER SENS
SUPPRESSION

Figure 3-7. Validyne differential pressure transducer.

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TABLE 3-14. CALIBRATION DATA FOR DIFFERENTIAL PRESSURE TRANSDUCER

Calibration Pressure (psi)	Transducer[a] Output (MV/V)
0	0.0
+0.005	49.0
+0.01	100.0
+0.005	49.3
0	0
-0.005	49.8
-0.01	100.0
-0.005	49.9
0	0

[a] Calibrated as a system with CD12, S/N 12677

Model No.	DP103
Serial No.	21300
Range NO.	±0.01 psi
Temp.	72°F
Date	7/8/76
Zero	608
Span	566

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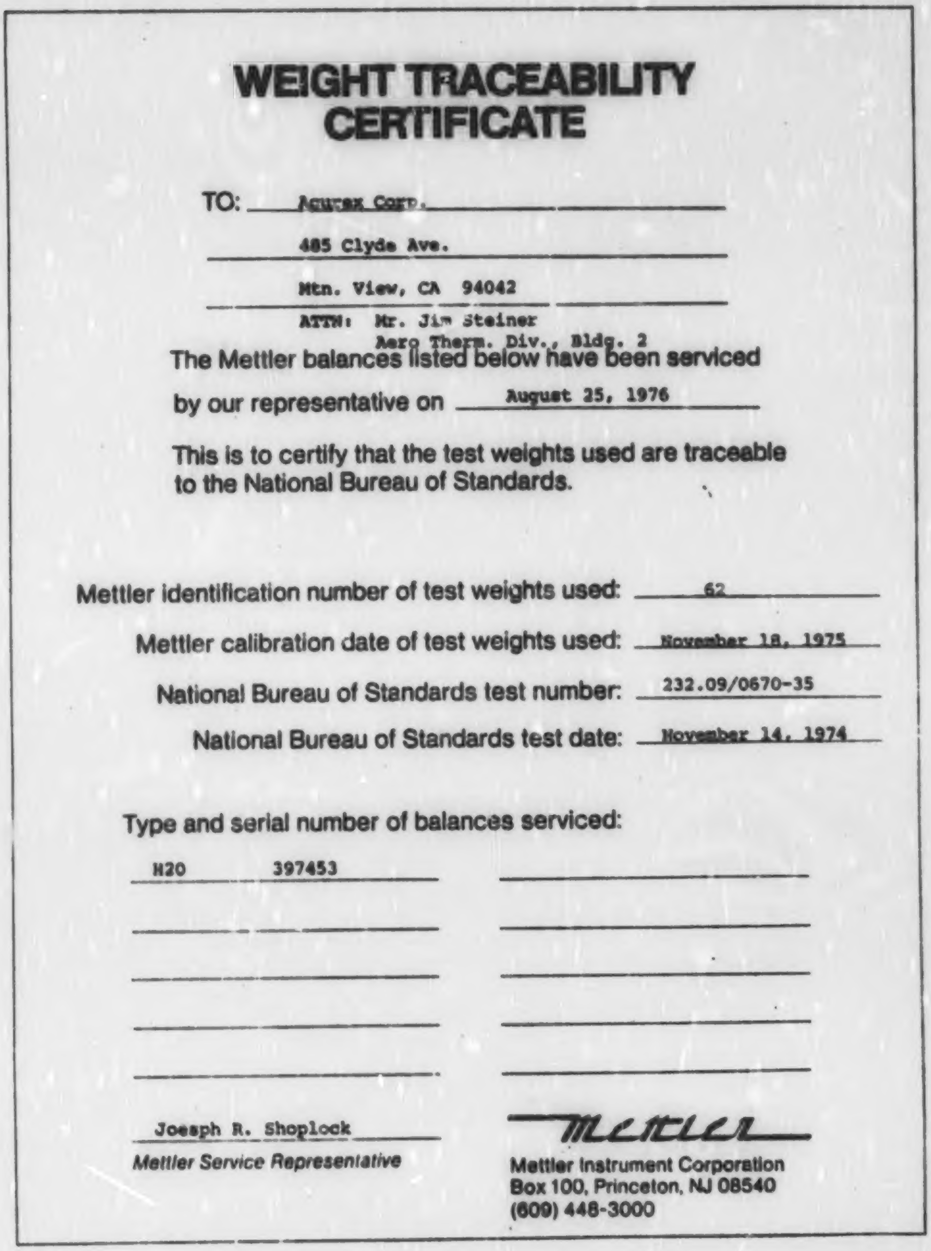
3.6 ISOPROPANOL

The Quality Assurance Branch of EPA recently conducted an evaluation of EPA Method 8 for the determination of sulfuric acid mist and sulfur dioxide emissions from stationary sources. They found that peroxide impurities in the isopropyl alcohol used for acid mist collection can convert sulfur dioxide to sulfuric acid and result in erroneously high acid mist values. To prevent this error, Aerotherm analyzed the isopropyl alcohol prior to shipping it into the field, in accordance with an EPA method, to determine if it contained any peroxides. Ten mls. of isopropyl alcohol were shaken with 10 mls. of freshly prepared 10 percent potassium iodide solution. A blank was prepared by similarly treating 10 mls. of distilled water, and the absorbance of each solution was read at 352 nanometers. The absorbance of the solution was less than 0.1, demonstrating that the alcohol was uncontaminated by peroxides.

3.7 METTLER ANALYTICAL BALANCE

A Mettler Model H20 balance, which can be read to 0.01 mg, was used to weigh the glass fiber filters and the chemicals used to prepare solutions. This balance is checked twice a year by the Mettler Instrument Corporation against National Bureau of Standard weights to insure its accuracy. Figure 3-8 illustrates the certificate issued by Mettler for the most recent calibration on the balance.



WEIGHT TRACEABILITY CERTIFICATE

TO: Acurex Corp.
485 Clyde Ave.
Mtn. View, CA 94042
ATTN: Mr. Jim Steiner
Aero Therm. Div., Bldg. 2

The Mettler balances listed below have been serviced by our representative on August 25, 1976

This is to certify that the test weights used are traceable to the National Bureau of Standards.

Mettler identification number of test weights used: 62

Mettler calibration date of test weights used: November 18, 1975

National Bureau of Standards test number: 232.09/0670-35

National Bureau of Standards test date: November 14, 1974

Type and serial number of balances serviced:

H20 397453

Joesph R. Shoplock
Mettler Service Representative

Mettler
Mettler Instrument Corporation
Box 100, Princeton, NJ 08540
(609) 448-3000

F124(5/74)

Figure 3-8. Weight traceability certificate.

SECTION 4

SAMPLING EQUIPMENT

This section describes the sampling equipment used by Aerotherm personnel for the three test locations (acid plant outlet duct, outlet duct from the reverb furnace electrostatic precipitator, and main smelter stack) at the Phelps-Dodge smelter. Also included is a description of modifications to the equipment required because of the physical constraints at each test location.

4.1 SO_3/H_2SO_4, SO_2, AND PARTICULATE SAMPLING EQUIPMENT

EPA Region IX specified that all compliance tests must use a combined EPA Method 5 and Method 8 sampling train. To meet this requirement, the High Volume Stack Sampler (HVSS) manufactured by Aerotherm was used for all SO_3/H_2SO_4, SO_2, and particulate sampling at Ajo. In its normal operating configuration, the HVSS complies with EPA Method 5 requirements and uses:

- A 316 stainless steel sampling nozzle properly sized for isokinetic sampling
- A 316 stainless steel lined sampling probe with an integral heater, a thermocouple to measure probe temperature, a thermocouple to measure stack gas temperature, and an S-type pitot tube to measure velocity pressure
- A 316 stainless steel cyclone to collect large particles and prevent premature plugging of the filter
- A teflon coated 304 stainless steel filter holder containing a 142 mm glass fiber filter
- A closed loop temperature controlled oven equipped with a thermocouple to indicate oven temperature
- A set of Lexan impingers in an icebath to remove moisture
- A 10 cfm carbon vane pump modified for very low leakage around the shaft
- A control console to monitor temperature, pressure and flowrates throughout the sampling train.

The console contains a Rockwell Model 415 dry gas meter which can accurately measure the volume of gas sampled to the nearest 0.005 ft^3 and an orifice meter to measure the instantaneous flowrate in the sampling train.

In order to bring the HVSS into line with the requirement that all compliance tests must use a combined EPA Method 5 and 8 sampling train, a number of modifications were made to the basic HVSS. These modifications are described in the following subsections.

4.1.1 Sampling Probes

For sampling particulates, SO_3/H_2SO_4, and SO_2, the sampling probes for the sampling trains were equipped with pyrex glass liners instead of the regular stainless steel, to prevent linear damage from acid gas and to eliminate any possibility of contaminating the particulate sample with metals leached from the steel liner. Stainless steel probe liners were used in the sampling trains which measured the moisture content and gas composition at the various test locations.

A special, extended sampling probe was assembled in the field to handle a peculiar situation encountered at the outlet duct of the acid plant. This duct was 4 feet in diameter, so a 5-foot probe could easily traverse the duct. However, the sampling port extensions were approximately 20 inches long, instead of the normal 4 inches to 6 inches. An extension was fabricated from 316 stainless steel to increase the length of the 5-foot probe so that all sampling points on a traverse could be used. The extension, shown in Figure 4-1, was also designed to position the sampling nozzle adjacent to the tips of the S-type pitot tube. EPA Region IX also requested that Aerotherm perform several comparisons using an in-stack filter and the EPA Method 5 out-of-stack filter. Figure 4-2 shows the in-stack filter holder assembly, mounted on the end of the sampling probe. The filter holder was made of 316 stainless steel and contained a 47 mm glass fiber filter, the same type of filter used in the out-of-stack filter holder in the heated oven. Care was taken to make certain the sampling nozzle was adjacent to the S-type pitot tube tips.

4.1.2 Heated Oven

The rectangular heated oven shown in Figure 4-3 was used at the acid plant outlet and the reverb furnace electrostatic precipitator outlet. This heated oven could not be used at the main smelter stack sampling site, because the existing unirail support system and the windows of the platform enclosure could not accommodate it. To overcome these problems, a smaller heated oven shown in Figure 4-4 was used. The smaller oven is circular, but has the same basic closed loop temperature control and thermocouple to measure oven temperature as the larger oven. Using this smaller oven also simplified the transfer of the sampling equipment from port to port at the main smelter stack.

4.1.3 Cyclone

Since no large particles or heavy grain loadings were expected at any of the three test locations, no cyclones were used in the sampling trains. The filter holder in the oven was connected directly to the glass lined sampling probe.

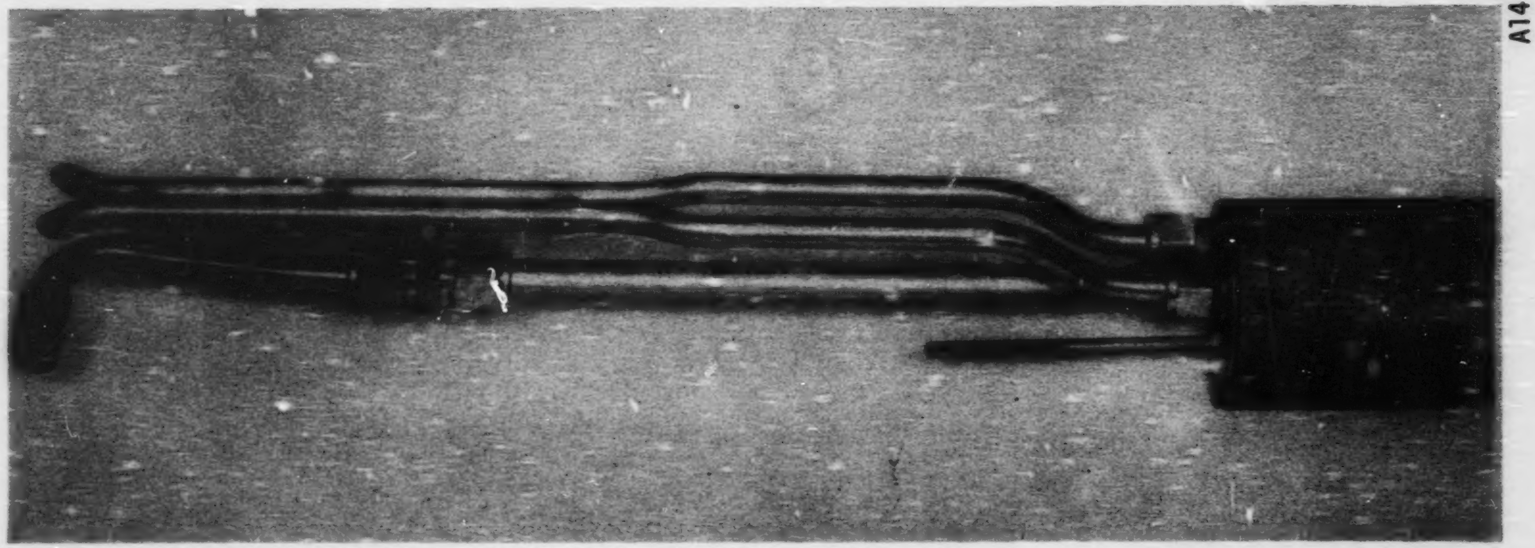


Figure 4-1. Extended sampling probe used at the acid plant outlet duct.

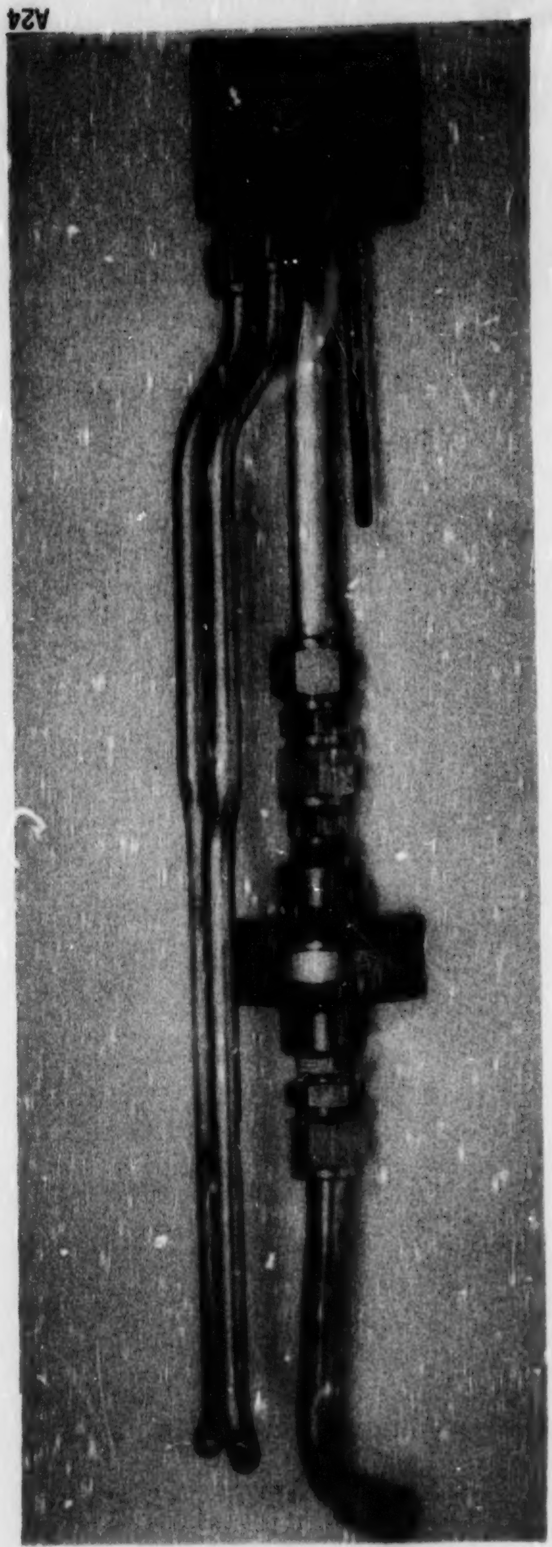


Figure 4-2. In-stack filter holder assembly.

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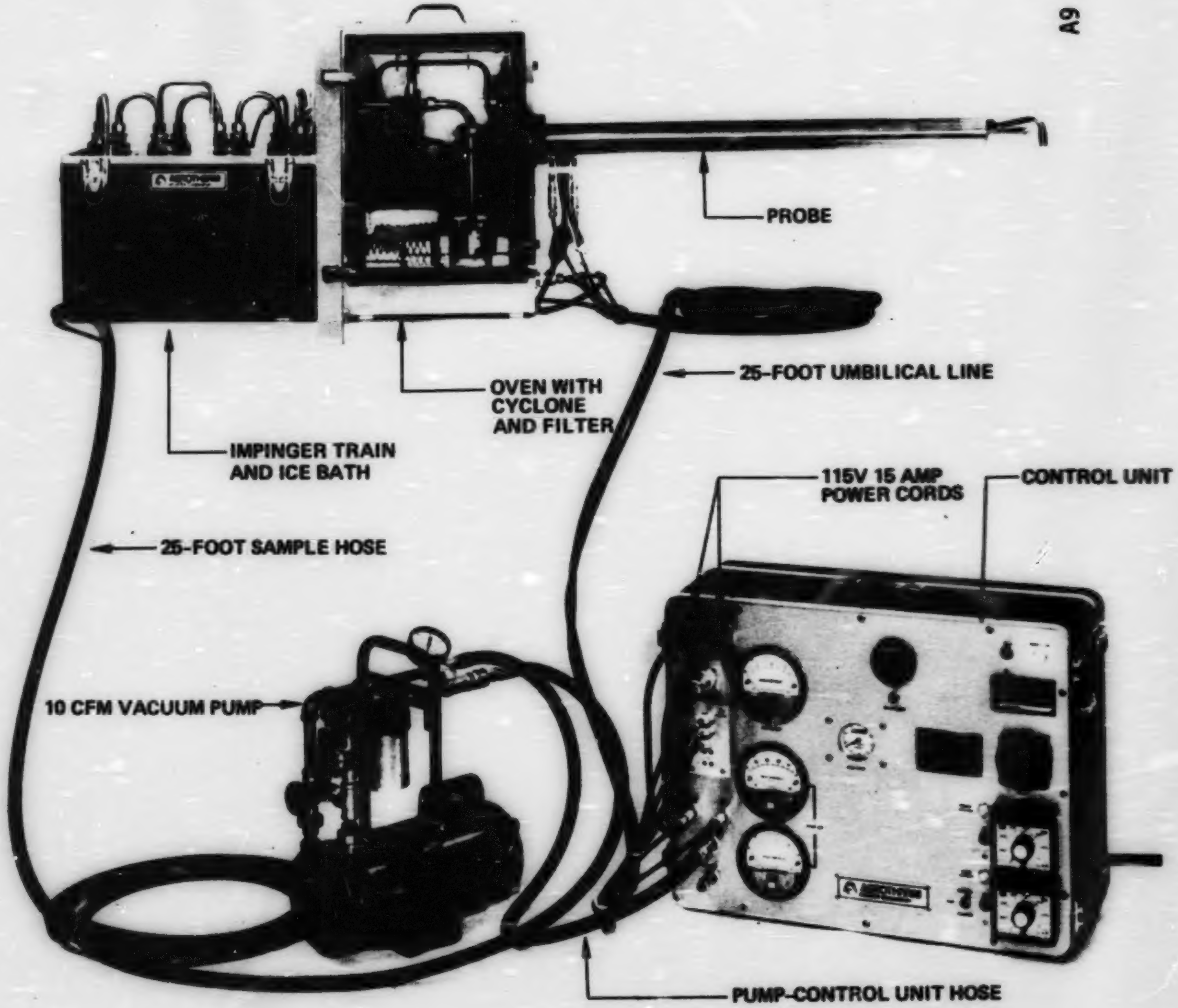
PROBE
25-FOOT UMBILICAL LINE
OVEN WITH CYCLONE AND FILTER
IMPINGER TRAIN AND ICE BATH
115V 15 AMP POWER CORDS
CONTROL UNIT
25-FOOT SAMPLE HOSE
10 CFM VACUUM PUMP
PUMP-CONTROL UNIT HOSE

Figure 4-3. Aerotherm high volume stack sampler.

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A12

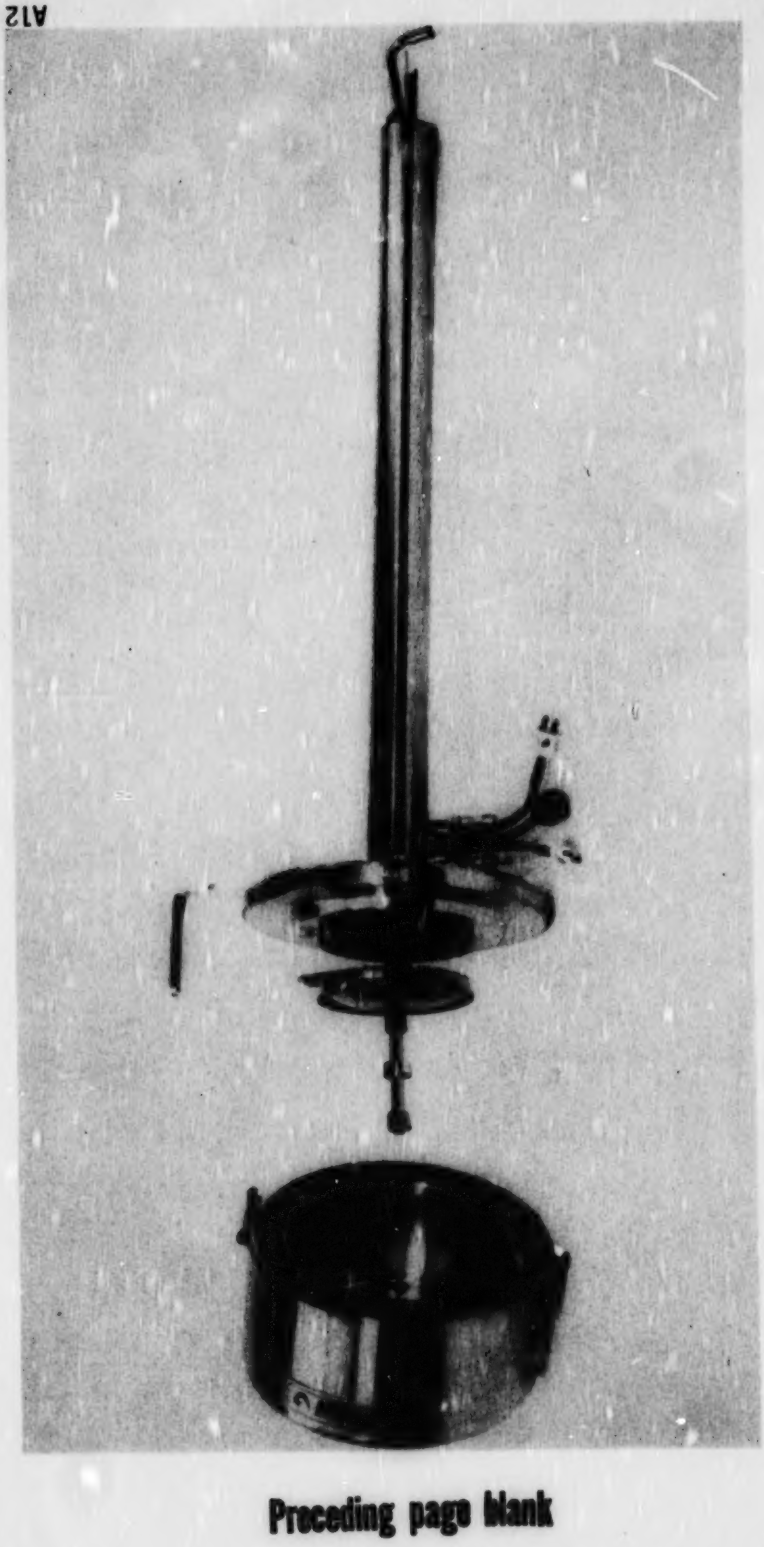

Figure 4-4. Small circular heated oven used at main smelter stack.

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4.1.4 Filter Holder

The 142mm filter holder used in the Aerotherm HVSS is shown at the right of Figure 4-5. (The left of the figure shows a filter holder used to sample the gases from the reverb furnace ESP outlet). This 142 mm filter holder is teflon coated on the front and back half internal surfaces to prevent the glass-fiber filter paper from sticking to the holder. This teflon coating holder also prevents acid from attacking the stainless steel holder and makes sample recovery easier. The filter itself is supported by a stainless steel screen to prevent it from being torn while the gas sample passes through it.

As the tests were being conducted, a problem developed with the teflon coating on the filter holder. An unknown component in the gas stream caused the teflon coating to flake off. A new filter holder was ordered from Mountain View to replace the original, which possibly had a defective coating, but the teflon coating again flaked off. These same holders were used for the duration of the test program. (A further discussion of this problem is found in Section 6.)

4.1.5 Impinger Train

The high flowrate impinger train normally used with the HVSS was replaced with the 1 cfm Greenburg-Smith impinger train shown in Figure 4-6. The first two impingers (1 bubbler, 1 standard tip) contained an 80 percent solution of isopropanol (IPA) to absorb SO_3/H_2SO_4. To capture any acid mist which might be entrained due to vigorous bubbling in the standard tip impinger, a glass filter holder containing a 50 mm glass fiber filter supported on a glass frit especially equipped with silicone rubber gaskets was used. Although EPA Method 8 only requires one IPA impinger to collect SO_3/H_2SO_4, a second impinger was added because 80 percent IPA solution is volatile and tends to evaporate rapidly. The use of a second impinger insured that even if evaporation did occur, there would be sufficient IPA solution present to quantitatively absorb the SO_3/H_2SO_4.

The third and fourth impingers (1 bubbler, 1 standard tip) contained 3 percent H_2O_2 solution (20 percent H_2O_2 in the case of the reverb furnace ESP outlet) to absorb the SO_2 passing through the IPA solution. A blank impinger (not required by EPA Method 8) was inserted between the fourth and sixth impingers as a precautionary measure, since occasionally the silica gel can back up if the vacuum in the sampling train is released too quickly during a leak test. If backup occurs, the blank impinger captures the silica gel and prevents it from contaminating the 3 percent H_2O_2 solution. The last (sixth) impinger contains silica gel to remove any moisture in the gases which has not condensed in the previous impingers. A thermocouple measures the temperature of the gases leaving the sixth impinger.

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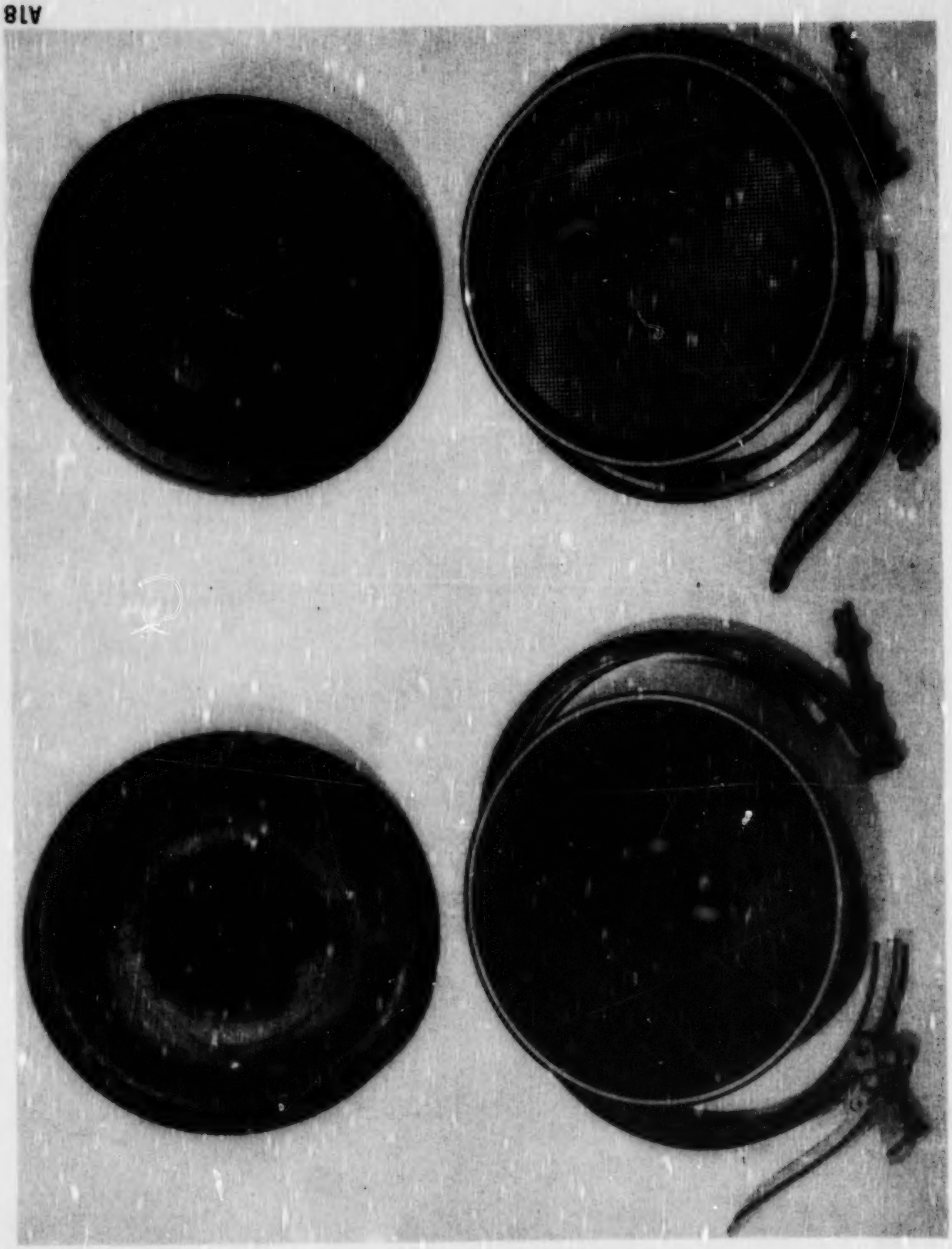

Figure 4-5. Comparison of new and used 142 mm filter holders.

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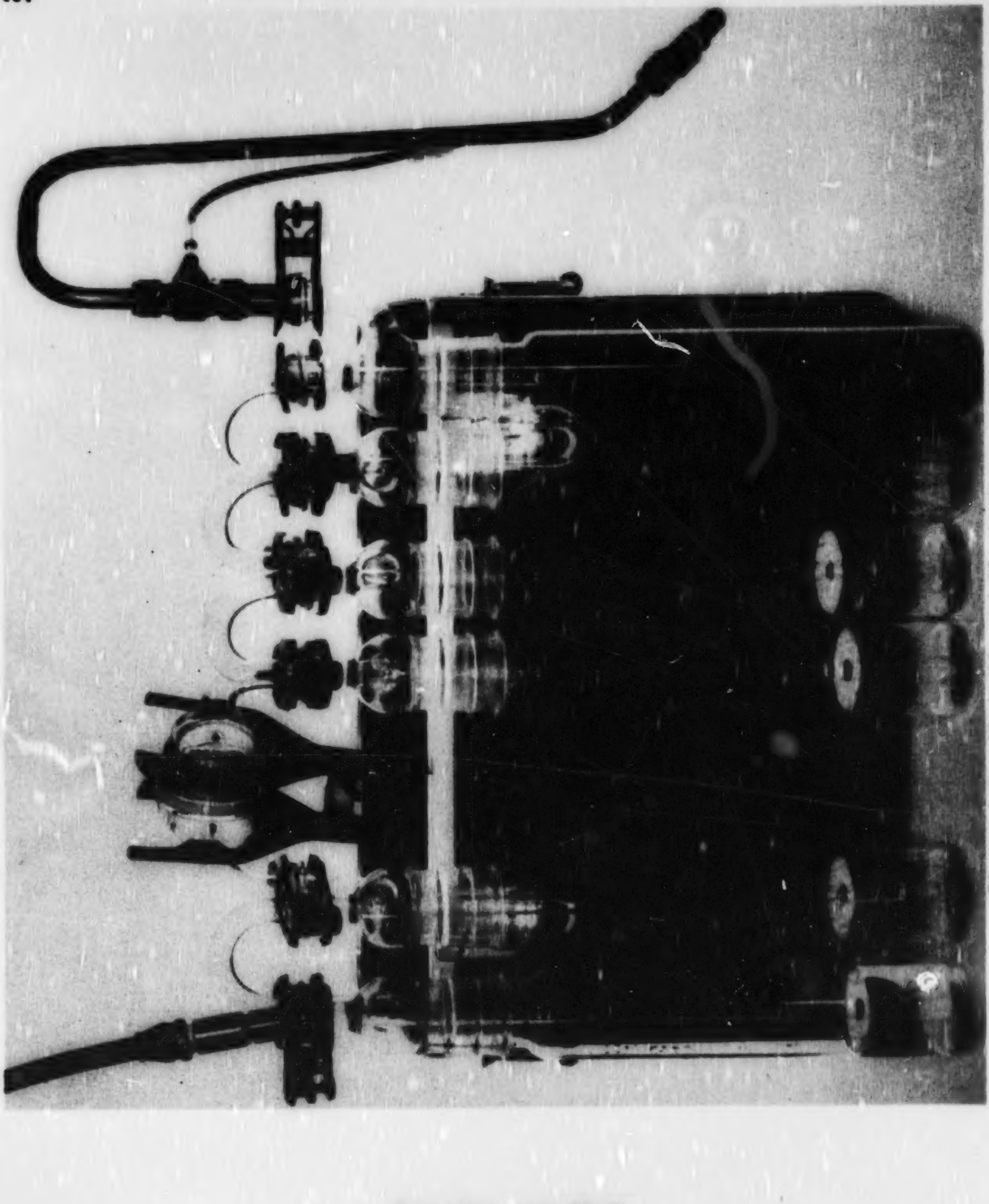

Figure 4-6. SO_3/H_2SO_4 and SO_2 impinger train.

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4.1.6 Control Console

Figure 4-7 depicts the control console for the HVSS used to monitor temperature, pressure, and flowrates throughout the sampling train. The control console is equipped with dual magnehelic gauges to indicate the velocity pressure sensed by the S-type pitot tube. However, during the tests on the main smelter stack when the fugitive gas collection system was off, the velocity in the stack was too low to be accurately indicated by the 0 to 0.5-inch magnehelic gage. When this occurred, the differential pressure transducer-analog indicator, previously illustrated in Figure 3-7, was used to indicate velocity pressure. This was the only modification to the control console.

With the modifications described above, the sampling trains used to measure the particulate, SO_3/H_2SO_4, and SO_2 content of the gases at the three test locations met or exceeded the requirements of EPA Methods 5 and 8 as published in the Federal Register.

4.2 STACK GAS COMPOSITION AND MOISTURE SAMPLING EQUIPMENT

Since EPA Region IX requested the use of EPA Method 8 in the back half of the particulate sampling trains, a separate sampling train was required to measure the stack gas composition and moisture content according to EPA Methods 3 and 4. This separate train was necessary because the isopropanol solution in the Method 8 impinger train evaporates during a test. This means that, although moisture does condense in the train, an erroneous result is obtained by measuring the volume of solution in the train because of the loss of IPA.

Figure 4-8 illustrates the combined EPA Method 3 and 4 sampling train used to measure stack gas composition and moisture content of the gases emitted from the main smelter stack and the reverb furnace ESP outlet. In this sampling train, a sample of the stack gas is extracted from the duct in proportion to the stack gas velocity. The particulates are then filtered out and the clean gases are split into 2 streams. One stream is measured for moisture in the Lexan impinger train immersed in the icebath. (This Lexan impinger train contains distilled water in the first two impingers, a blank in the third, and silica gel in the fourth impinger.) The second gas stream is pumped through a rotameter to a tedlar bag. After the moisture has been removed, the gases are pumped to a control console to measure and record the necessary temperature and volume data.

4.3 STACK GAS COMPOSITION

At the acid plant, only a stack gas composition measurement was made, since there is no moisture in the exit gases from an acid plant. This sampling train for this test consisted of a sampling nozzle, an in-stack 47 mm filter holder, a stainless steel tube with an attached S-type pitot tube,

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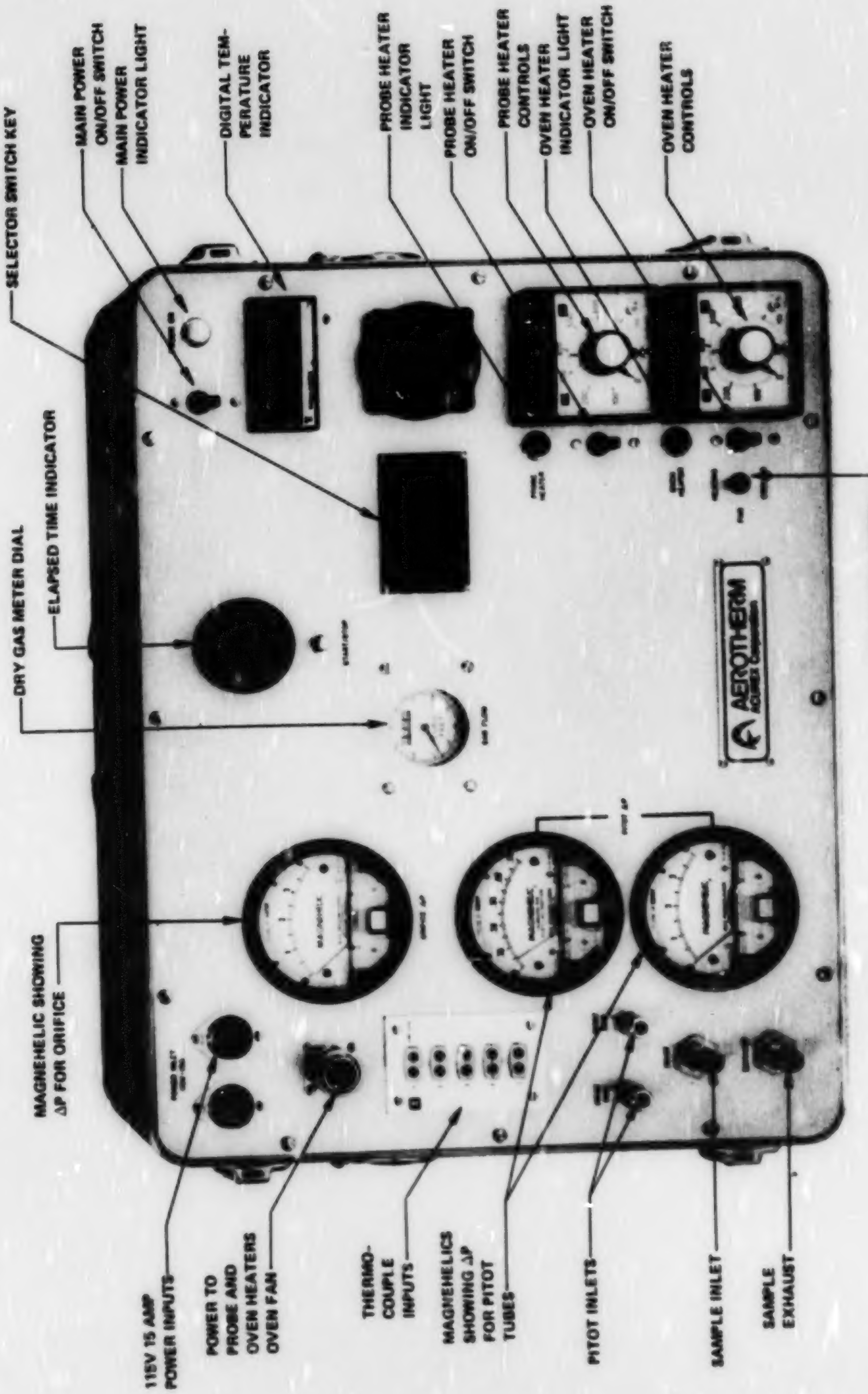
SELECTOR SWITCH KEY
MAIN POWER ON/OFF SWITCH
MAIN POWER INDICATOR LIGHT
DIGITAL TEMPERATURE INDICATOR
PROBE HEATER INDICATOR LIGHT
PROBE HEATER ON/OFF SWITCH
PROBE HEATER CONTROLS
OVEN HEATER INDICATOR LIGHT
OVEN HEATER ON/OFF SWITCH
OVEN HEATER CONTROLS
FAN ON/OFF SWITCH
DRY GAS METER DIAL
ELAPSED TIME INDICATOR
AEROTHERM
MAGNEHELIC SHOWING ΔP FOR ORIFICE
115V 15 AMP POWER INPUTS
POWER TO PROBE AND OVEN HEATERS OVEN FAN
THERMOCOUPLE INPUTS
MAGNEHELICS SHOWING ΔP FOR PITOT TUBES
PITOT INLETS
SAMPLE INLET
SAMPLE EXHAUST

Figure 4-7. Control console for HVSS.

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Figure 4-8. Combined EPA Method 3 and 4 sampling train.

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a tygon line, a diaphragm pump, a rotometer, and a tedlar bag. The contents of a tedlar bag sample were analyzed using a conventional Orsat analyzer. No differentiation was made between the SO_2 and CO_2.

The following section describes how the sampling equipment was used in the field to obtain the necessary emission data.

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SECTION 5

SAMPLING PROCEDURES

This section describes the emissions sampling procedures used by Aerotherm personnel at all three test locations. During the test program, situations naturally arose which required modifications to these test procedures. These modifications are discussed in Appendix A of this report.

5.1 PRELIMINARY MEASUREMENTS

Before conducting a stack test according the EPA Method 5, a series of preliminary measurements must be made using EPA Methods 1, 2, 3, and 4 to determine:

- The location of the sampling site and the number of sampling points to be used (EPA Method 1)
- The velocity, temperature, pressure, and volumetric flowrate of the gases at the sampling site (EPA Method 2)
- The composition of the gases to be sampled (EPA Method 3)
- The moisture content of the gases to be sampled (EPA Method 4)

These and other preliminary measurements were made simultaneously at all three test locations. A description of the procedures used at each test location follows.

5.1.1 Acid Plant Outlet Sampling Site

To calculate stack diameter, duct diameter plus the length of the sampling port was measured by inserting a rod into the duct through a sampling port until it hit the opposite side. The rod was then marked and measured. Next, the length of the sample port was directly measured using a metal tape. The diameter of the stack was then calculated by subtracting the length of the sample port from the stack diameter.

After calculating stack diameter, Aerotherm's test crew (Sutton, Densmore) then measured the upstream and downstream distances from flow disturbances to the sampling site. These measurements showed that the site was more than eight duct diameters from a downstream flow disturbance and more than two duct diameters from an upstream flow disturbance. Under these conditions, Table 1-1 of EPA Method 1 requires a minimum of 12 sampling points (6 on each of two diameters at 90° to each other). In accordance with these specifications, vertical and horizontal traverse positions at the acid plant outlet sampling site were marked on the sampling probe

Once these sampling point locations were marked on the sampling probe, the probe and oven were connected and hung on the unirail assembly in the horizontal traverse position (as shown in Figure 5-1).

Next, measurements were made using EPA Method 2. The quick disconnect fittings which connect the S-type pitot tube to the magnehelic gage in the control module via the umbilical cord were inspected to insure they were tight and leak-free, and the 0 to 4-inch pitot magnehelic gage in the control console was zeroed and leveled. After checking with the process observer stationed on the ground to make sure the acid plant was operating normally, the test crew carefully inserted the sampling probe (with nozzle, thermocouple, and S-type pitot tube) into the long sample port and positioned it at sampling point #1. The test crew made sure the S-type pitot tube tips were pointing directly into the gas streamlines, then packed the openings between the probe sheath and the sample port with packing paper to seal the probe in the port. Next, the velocity pressure and gas temperature were measured and recorded on the data sheet shown in Figure 5-2.

This measurement procedure was repeated at each of the six sampling points. Having completed the traverse, the probe was positioned with the S-type pitot tube tips approximately in the center of the duct and the openings parallel to the gas streamlines. The lower pressure side of the pitot tube was then disconnected and the static pressure in the duct was measured using the high pressure side. This measurement was also recorded on the data sheet and the probe removed from the duct.

Having completed the requirements of EPA Method 2, the test crew collected a sample of the stack gas for chemical analysis using EPA Method 3 and the sampling train described in Section 4.3. The Tedlar bag was inspected and checked for leaks, then evacuated and set up on a stand as shown in Figure 4-8. The sampling probe was placed at a point of average velocity in the stack and purged with the stack gas. The sample port was then sealed with packing paper and the bag connected to the sampling train.

After making sure the connections were tight and leak free, the test crew obtained an integrated sample of the stack gases by sampling in proportion to the stack gas velocity. Sampling data was recorded on the data sheet and — at the completion of the run — the Tedlar bag was taken to the laboratory room (provided by Phelps-Dodge) for analysis. An Orsat analyzer was used to measure the CO_2 (and SO_2), CO, and O_2 content of the bag, and these data were recorded on the data sheet shown in Figure 5-3. No moisture content measurements were made using EPA Method 4 since there is no moisture present in the effluent of a sulfuric acid plant.

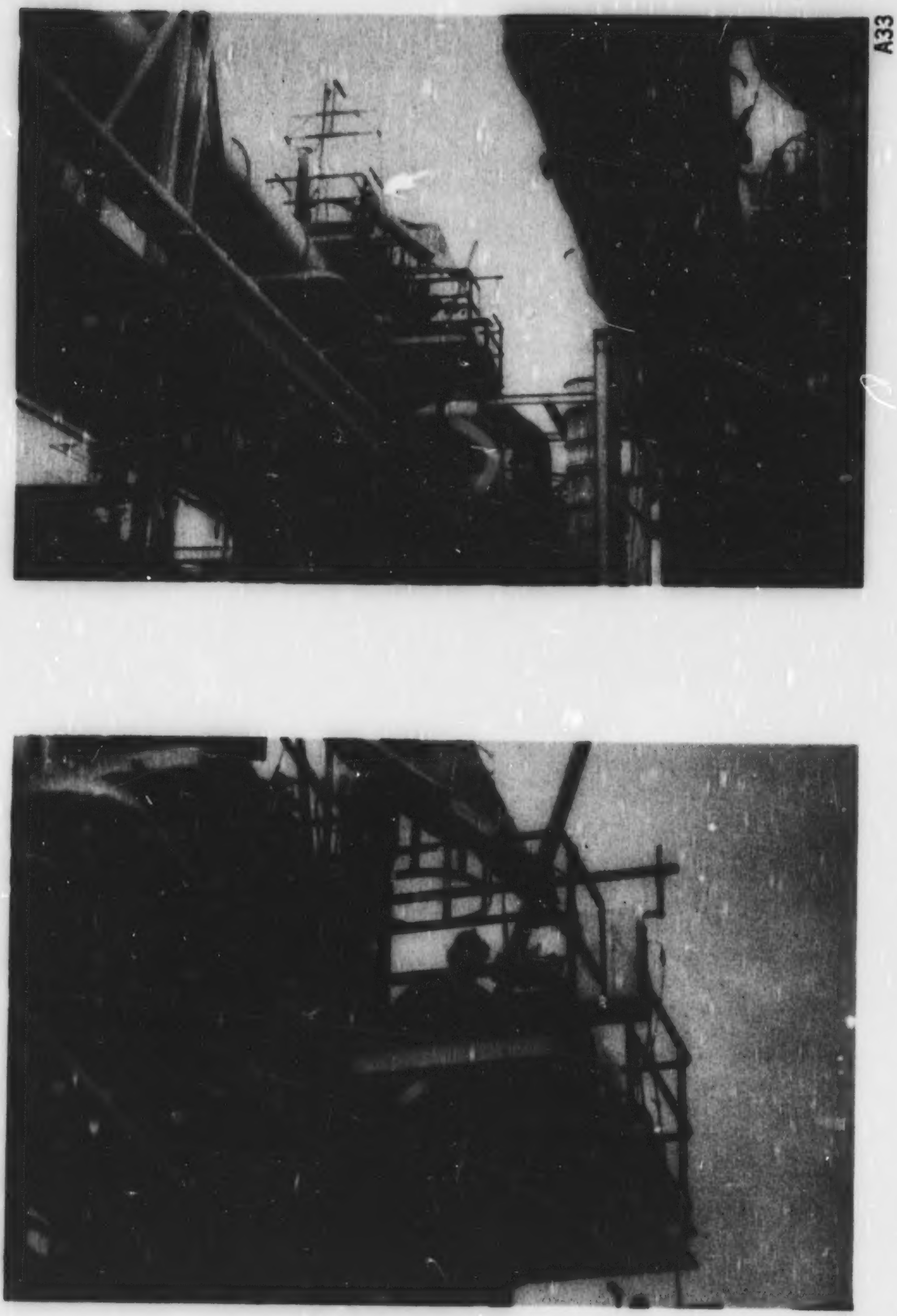

Figure 5-1. Acid plant outlet sampling site.

[illegible] PLANT
LOCATION
STACK DIAMETER (IN.)
DUCT DIMENSIONS (IN.) x (IN.)

3.01 DATE
TIME
RUN

3.02 OPERATORS

3.03 STATIC PRESSURE (IN. WG.)
AMBIENT PRESSURE (IN. HG.)
TYPE S PITOT COEFFICIENT

3.07 STACK PRESSURE (IN. HG.)
MOLECULAR WEIGHT (LB/LB-MOLE)
B_{wo}

VELOCITY TRAVERSE DATA

3.06

Sampling Point Number	Velocity Head (in. wg.)	$\sqrt{\Delta P}$	Stack Gas Temp (°F)	Sampling Point Velocity (fps)
		Average	Average	

EOF
EOF

Figure 5-2. Preliminary velocity traverse data sheet.

Preceding page blank

1 PLANT
LOCATION
STACK DIAMETER (IN.)
DUCT DIMENSIONS (IN.) x (IN.)

2,1 DATE
TIME
RUN

2,2 OPERATORS

2,3 STATIC PRESSURE (IN. HG.)
AMBIENT PRESSURE (IN. HG.)
TYPE S PITOT COEFFICIENT

STACK GAS MOLECULAR WEIGHT

2,5

Clock Time	Rotameter Reading (cfh)	Velocity Head (in. wg.)	Stack Gas Temp (°F)

EOF

2,6

CO_2 (% by Vol.)	O_2 (% by Vol.)	CO (% by Vol.)
Average	Average	Average

EOF
EOF

Figure 5-3. Stack gas composition data sheet.

5.1.2 Reverb Furnace Electrostatic Precipitator Outlet Sampling Site

At this test location, separate outlet ducts from two precipitators join and make a 90° turn into an ID fan located on the ground. The sampling site, shown in Figure 5-4, is located between [illegible]

At this location, duct diameter was measured with a sampling probe in place of the rod used by the acid plant crew. This was done to eliminate any error due to bending of the rod. The duct at this site was found to be 6 feet in diameter. The test crew (Knirck and Gilchrist) measured the distances to the downstream flow disturbance (y-joint to the sampling site) and to the upstream flow disturbance (sampling site to 90° elbow) and found these distances to be four and two duct diameters, respectively. Under these conditions, EPA Method 1 requires a total of 36 sampling points (18 on each of two horizontal traverses 90° apart). A-traverse was in the plane of the duct bend and B-traverse was perpendicular to the plane of the duct bend. Because the gas temperature was ~550°F to 600°F, a high temperature grease marker was used to mark the sampling point locations on the probe.

The velocity and temperature traverses (A and B ports) were performed as described for the acid plant, except that the 3-inch ID sample ports were not sealed with paper packing because of the high gas temperature.

The composition and moisture content of the stack gases were measured using the combined EPA Method 3 and 4 sampling train. The combined sampling train, shown in Figure 4-8, consists of a sampling nozzle, a 5-foot heated probe (300°F to 325°F) with a stainless steel liner and an S-type pitot tube, a 142 mm filter holder (unheated) attached to the end of the probe, and other components. Prior to this test, a measured volume of distilled water and a known weight of silica gel had been put into the Lexan impinger train in the laboratory. At the sampling site, the sampling train was assembled and the moisture portion of the train was checked for leaks at 15 inches Hg vacuum by plugging the probe inlet (using a blank swagelok) and the tee connection to the Tedlar bag. The Tedlar bag was then leak checked separately, evacuated, and connected to the tee in the impinger train.

To make a run, the sampling probe was positioned at a point of average velocity, and the data were recorded on the data sheets shown in Figures 5-3 and 5-5. At the completion of the run, the equipment was disassembled and the impinger train, Tedlar bag, and 142 mm filter holder were taken to the laboratory for sample recovery and recording and analysis of data.

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Figure 5-4. Sampling site on reverb ESP outlet.

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0,0 [PLANT
LOCATION

0,1 [STACK DIAMETER (IN.)

0,2 [DUCT DIMENSIONS (IN.) x (IN.)

1,0

1,1 [DATE
TIME
RUN

1,2 [OPERATORS

BOF

1,3 [STATIC PRESSURE (IN. HG.)
AMBIENT PRESSURE (IN. HG.)
TYPE S PITOT COEFFICIENT

1,4 [WET BULB TEMP (°F)
DRY BULB TEMP (°F)
MOISTURE (% VOL.)
B_{wo}

STACK GAS MOISTURE (CONDENSATION)

1,5

	Impinger #1 Weight (gm)	Impinger #2 Weight (gm)	Silica Gel Weight (gm)
Final			
Initial			
Liquid Collected			
			Total, $V_{\ell c}$

EOF

1,6

Clock Time	Dry Gas Meter (ft³)	Rotameter Reading (cfh)	Gas Meter Temp (°F)	Velocity Head (in. wg.)	Stack Gas Temp (°F)
	Total, V_m	Average	Avg., T_m		

EOF
EOF

Figure 5-5. Stack gas moisture sheet.

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5.1.3 Main Smelter Stack

The sampling site on the main smelter stack is shown in Figure 5-6. Since the stack diameter was large, a rod or probe could not be used to measure the diameter. Instead, the test crew (Stanley and Rice) measured the circumference of the stack at the level of the sampling ports and the wall thickness of the stack at ports A, B, and C to compute the inside stack diameter of 253 inches or 21 feet. The distance to the downstream flow disturbance, found to be approximately four stack diameters, was measured by lowering a rope with a weight on the end over the side of the sampling platform until the weight just reached the top of the breeching that enters the base of the stack. The upstream flow disturbance (stack exit) was found to be much greater than two stack diameters away. Under these conditions, EPA Method 1 requires a total of 36 sampling points (12 on each of three horizontal traverses, as shown in Figure 5-7). The sampling point locations were marked on the sampling probe using duct tape. It should be noted that, although it was physically impossible to sample on two perpendicular diameters, the use of three radii provided accurate results since the three radii were located in the plane of maximum variation of particulate and gas flow.

The velocity and temperature at ports A, B, and C were measured as at the other sites and the sample ports were sealed with paper packing. Nothing could be done, however, to prevent considerable leakage of air around the sample port plate in port B, since the sample port itself had deteriorated.

The composition and moisture content of the stack gases were measured using sample procedures and equipment as described for the reverb furnace ESP outlet, except that a 47 mm filter holder was used in place of the 142 mm filter holder.

5.2 PRELIMINARY CALCULATIONS

After preliminary data had been gathered as described, a series of calculations was made at each test location to determine:

- Dry molecular weight (M_d) of the stack gases using Equation (3-2) of the Federal Register
- Wet molecular weight (M_s) of the stack gases using $M_s = M_d (1 - B_{wo}) + 18 (B_{wo})$
- Moisture content of the stack gases (B_{wo}) using Equations (4-1), (4-2), and (4-3) of the Federal Register.

These terms (M_d, M_s, B_{wo}) are assumed to remain constant during a given test run. Although variations in M_d and M_s have an insignificant effect on isokinetic sampling, variations in B_{wo} will

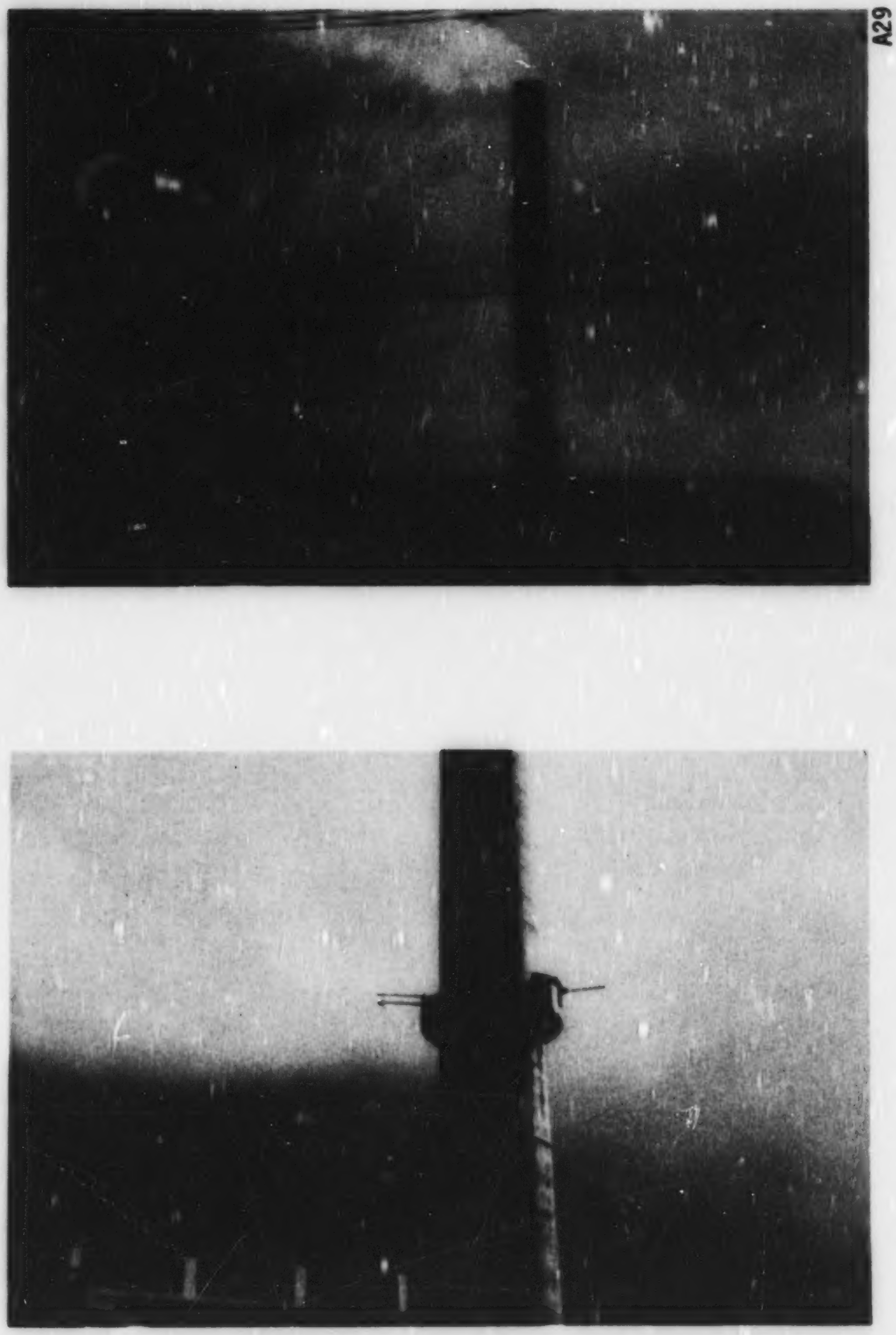
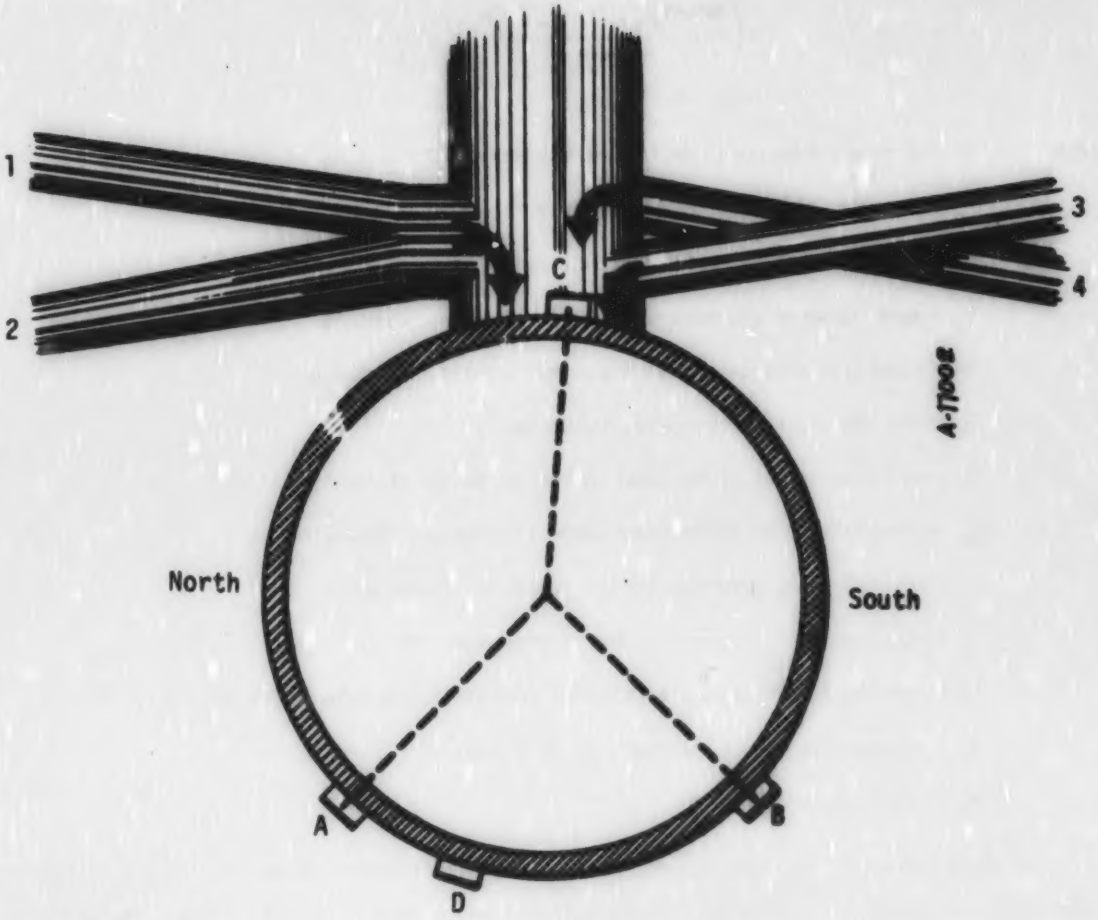


Figure 5-6. Sampling site on main smelter stack.


1
2
3
4
C
North
South
A
B
D
A-71008

1. Reverb gas (500°F)
2. Acid Plant tail gas (150°F)
3. Reverb smoke hood (70°F)
4. Converter smoke hood (200°F)

Figure 5-7. Location of sampling ports and inlet ducts on main smelter stack.

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have a significant effect. An examination of the following equation, used to calculate the flowrates required for isokinetic sampling, will illustrate this:

$$\Delta H = \left(\frac{60^2\pi^2 N_d^4 K_p^2 C_v^2 (1 - B_{wo})^2 P_s M_d}{576^2 K_o^2 M_s P_m}\right)\left(\frac{T_m}{T_s}\right)\Delta P$$

where ΔH = pressure drop across orifice meter, inches H_2O

K_p = constant, 85.48 (ft/sec)[(lb/lb mole °R)(in Hg/in H_2O)]$^{1/2}$

C_v = S-type pitot tube coefficient, dimensionless

T_s = absolute stack gas temperature at the sampling point, °R

ΔP = stack gas velocity head at the sampling point, inches H_2O

P_s = absolute stack gas pressure, inches Hg

M_s = molecular weight of the stack gas on wet basis, lb/lb-mole

B_{wo} = proportion by volume of water vapor in stack gas, dimensionless

P_m = absolute gas pressure at the orifice meter, inches Hg

T_m = absolute gas temperature at the orifice meter, °R

K_o = constant determined by calibration, (ft^3/min)[lb/lb mole °R)(in Hg/in H_2O)]$^{1/2}$

M_d = molecular weight of the stack gas on dry basis, lb/lb-mole

N_d = nozzle diameter, inches

Since the term $(1 - B_{wo})$ is squared, it has a significant effect on the isokinetic sampling rate (ΔH). For example, if the moisture content of the reverb furnace ESP outlet stack gas (determined to be 0.127 during the preliminary measurements), changes by 5 percent (a change in B_{wo} of 0.05), $(1 - B_{wo})$ will change from 0.76 to 0.67 — and ΔH will change by 11 percent. Changes in the moisture content of the stack gas cannot be predicted unless a preliminary moisture measurement is made prior to each test. However, this was not done due to time limitations imposed by the length of the copper blow.

To find the isokinetic sampling rate, the above equation is solved by inserting:

- Measured values (M_s, M_d, B_{wo}, T_m, P_m) determined during the preliminary measurements using Methods 3 and 4
- Calibration constants (C_v, K_o) determined in the laboratory

- Measured values (P_s, T_s, ΔP) determined using Method 2
- Various values of nozzle diameter (N_d).

This calculation also determines the size of sampling nozzle needed to sample isokinetically over the entire range of ΔP in a particular duct, at a rate which will satisfy criteria of 60 scf total volume of gas sampled in the 60 minutes available for sampling.

After a number of these calculations are completed, the equation is reduced to the following form:

$$\Delta H = K \Delta P$$

where K is a numerical constant. Having determined K, the operator of the control module prepares a table of values of ΔP versus ΔH for use during the actual stack test. Then, during a test the operator reads the ΔP measured by the S-type pitot tube, finds the corresponding value of ΔH required for isokinetic sampling from the table, and adjusts the suction on the pump to provide the required ΔH across the orifice meter.

Once the preliminary measurements and calculations have been completed, the actual stack tests can be conducted. Sampling procedures used during the actual test periods are described below.

5.3 EPA METHOD 5 AND 8 FIELD TEST PROCEDURES

The sampling procedure used at each test location (shown in Figures 5-8, 5-9, and 5-10) consist of three separate steps: preparation, use, and disassembly of the sampling train. Each step is described in detail in the following sections.

5.3.1 Preparation of the Sampling Trains

All sampling train preparation was done in the clean laboratory room provided by Phelps-Dodge to minimize the chances of contaminating the equipment prior to the field test. Analytical reagent grade chemicals were used to prepare the IPA and H_2O_2 solutions, with one exception that occurred during the early stages of the test program. Reagent grade H_2O_2 (30 percent), shipped by truck to Ajo in advance of the test crew, was destroyed during transit. H_2O_2 was reordered and shipped a second time to Ajo by truck -- but the chemicals did not arrive at the smelter until July 22, 1976. Thus, tests #1, #2, and #3 at each test location used 3 percent and 20 percent H_2O_2 solution provided by Phelps-Dodge. Although the source of the H_2O_2, its age or quality (analytical reagent versus technical grade), could not be determined, Phelps-Dodge personnel had been using the H_2O_2 and had found it to be adequate. (A solution of 20 percent H_2O_2 was used at the reverb test location

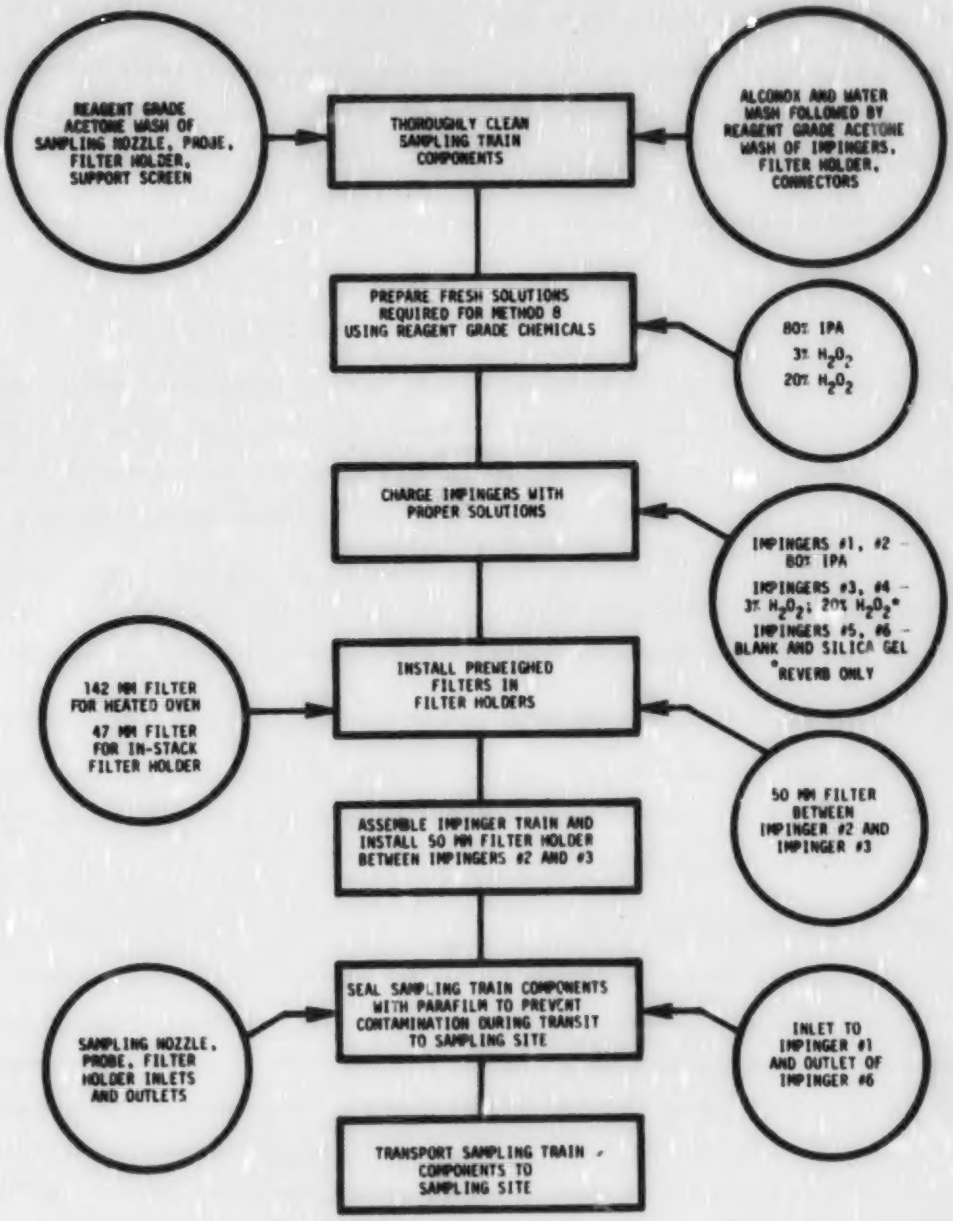
REAGENT GRADE ACETONE WASH OF SAMPLING NOZZLE, PROBE, FILTER HOLDER, SUPPORT SCREEN
THOROUGHLY CLEAN SAMPLING TRAIN COMPONENTS
ALCONOX AND WATER WASH FOLLOWED BY REAGENT GRADE ACETONE WASH OF IMPINGERS, FILTER HOLDER, CONNECTORS
PREPARE FRESH SOLUTIONS REQUIRED FOR METHOD 8 USING REAGENT GRADE CHEMICALS
80% IPA
3% H_2O_2
20% H_2O_2
CHARGE IMPINGERS WITH PROPER SOLUTIONS
IMPINGERS #1, #2 - 80% IPA
IMPINGERS #3, #4 - 3% H_2O_2; 20% H_2O_2*
IMPINGERS #5, #6 - BLANK AND SILICA GEL
*REVERB ONLY
142 MM FILTER FOR HEATED OVEN
47 MM FILTER FOR IN-STACK FILTER HOLDER
INSTALL PREWEIGHED FILTERS IN FILTER HOLDERS
50 MM FILTER BETWEEN IMPINGER #2 AND IMPINGER #3
ASSEMBLE IMPINGER TRAIN AND INSTALL 50 MM FILTER HOLDER BETWEEN IMPINGERS #2 AND #3
SAMPLING NOZZLE, PROBE, FILTER HOLDER INLETS AND OUTLETS
SEAL SAMPLING TRAIN COMPONENTS WITH PARAFILM TO PREVENT CONTAMINATION DURING TRANSIT TO SAMPLING SITE
INLET TO IMPINGER #1 AND OUTLET OF IMPINGER #6
TRANSPORT SAMPLING TRAIN COMPONENTS TO SAMPLING SITE

Figure 5-8. EPA Method 5 and 8 sampling train preparation flow chart.

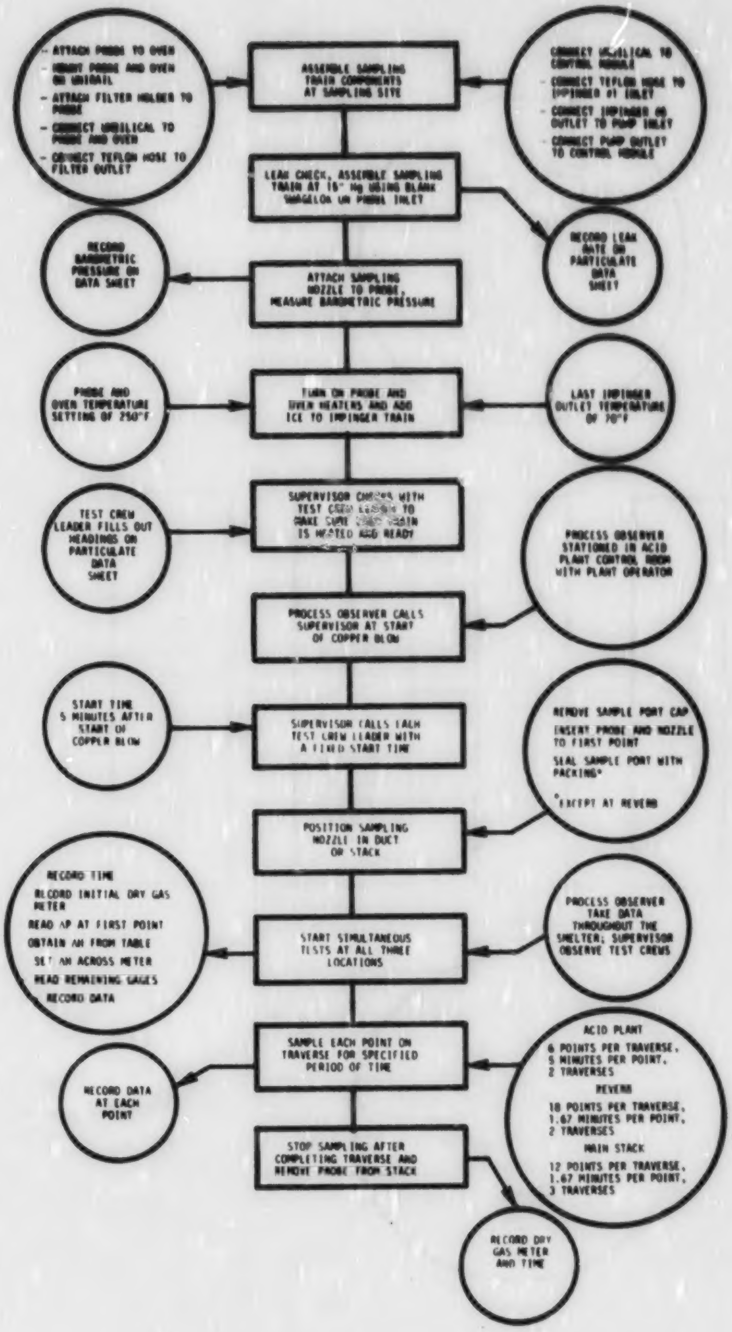
- ATTACH PROBE TO OVEN
- MOUNT PROBE AND OVEN ON UNIRAIL
- ATTACH FILTER HOLDER TO PROBE
- CONNECT UMBILICAL TO PROBE AND OVEN
- CONNECT TEFLON HOSE TO FILTER OUTLET
ASSEMBLE SAMPLING TRAIN COMPONENTS AT SAMPLING SITE
CONNECT UMBILICAL TO CONTROL MODULE
- CONNECT TEFLON HOSE TO IMPINGER #1 INLET
- CONNECT IMPINGER #4 OUTLET TO PUMP INLET
- CONNECT PUMP OUTLET TO CONTROL MODULE
LEAK CHECK, ASSEMBLE SAMPLING TRAIN AT 15" Hg USING BLANK SWAGELOK ON PROBE INLET
RECORD LEAK RATE ON PARTICULATE DATA SHEET
RECORD BAROMETRIC PRESSURE ON DATA SHEET
ATTACH SAMPLING NOZZLE TO PROBE, MEASURE BAROMETRIC PRESSURE
PROBE AND OVEN TEMPERATURE SETTING OF 250°F
TURN ON PROBE AND OVEN HEATERS AND ADD ICE TO IMPINGER TRAIN
LAST IMPINGER OUTLET TEMPERATURE OF 70°F
TEST CREW LEADER FILLS OUT HEADINGS ON PARTICULATE DATA SHEET
SUPERVISOR CHECKS WITH TEST CREW LEADER TO MAKE SURE THAT TRAIN IS HEATED AND READY
PROCESS OBSERVER STATIONED IN ACID PLANT CONTROL ROOM WITH PLANT OPERATOR
PROCESS OBSERVER CALLS SUPERVISOR AT START OF COPPER BLOW
START TIME 5 MINUTES AFTER START OF COPPER BLOW
SUPERVISOR CALLS EACH TEST CREW LEADER WITH A FIXED START TIME
REMOVE SAMPLE PORT CAP
INSERT PROBE AND NOZZLE TO FIRST POINT
SEAL SAMPLE PORT WITH PACKING*
*EXCEPT AT REVERB
POSITION SAMPLING NOZZLE IN DUCT OR STACK
RECORD TIME
RECORD INITIAL DRY GAS METER
READ ΔP AT FIRST POINT
OBTAIN ΔH FROM TABLE
SET ΔH ACROSS METER
READ REMAINING GAGES
RECORD DATA
START SIMULTANEOUS TESTS AT ALL THREE LOCATIONS
PROCESS OBSERVER TAKE DATA THROUGHOUT THE SMELTER; SUPERVISOR OBSERVE TEST CREWS
SAMPLE EACH POINT ON TRAVERSE FOR SPECIFIED PERIOD OF TIME
RECORD DATA AT EACH POINT
ACID PLANT
6 POINTS PER TRAVERSE, 5 MINUTES PER POINT, 2 TRAVERSES
REVERB
10 POINTS PER TRAVERSE, 1.67 MINUTES PER POINT, 2 TRAVERSES
MAIN STACK
12 POINTS PER TRAVERSE, 1.67 MINUTES PER POINT, 3 TRAVERSES
STOP SAMPLING AFTER COMPLETING TRAVERSE AND REMOVE PROBE FROM STACK
RECORD DRY GAS METER AND TIME

Figure 5-9. EPA Method 5 and 8 sampling procedure flow chart.

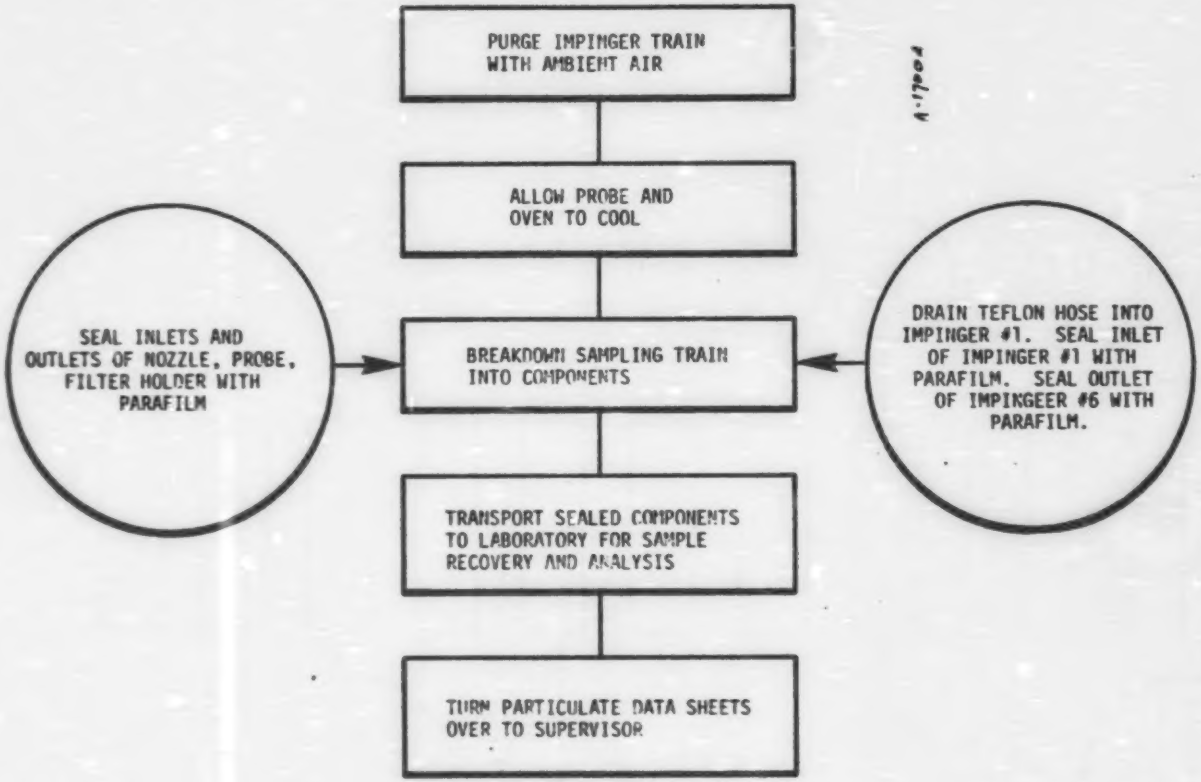
PURGE IMPINGER TRAIN WITH AMBIENT AIR
ALLOW PROBE AND OVEN TO COOL
SEAL INLETS AND OUTLETS OF NOZZLE, PROBE, FILTER HOLDER WITH PARAFILM
BREAKDOWN SAMPLING TRAIN INTO COMPONENTS
DRAIN TEFLON HOSE INTO IMPINGER #1. SEAL INLET OF IMPINGER #1 WITH PARAFILM. SEAL OUTLET OF IMPINGEER #6 WITH PARAFILM.
TRANSPORT SEALED COMPONENTS TO LABORATORY FOR SAMPLE RECOVERY AND ANALYSIS
TURN PARTICULATE DATA SHEETS OVER TO SUPERVISOR

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Figure 10. EPA Method 5 and 8 sampling train breakdown flowchart.

to insure that the absorption solution would not be consumed by the high concentrations of SO_2 present in the gases.)

To comply with EPA Method 8, all solutions (80 percent IPA, 3 percent H_2O, 20 percent H_2O) and rinsing solutions for sample recovery were prepared each day to insure they maintained their strength.

A final precautionary step taken was to seal the various inlets and outlets of the sampling train components with parafilm to prevent any foreign material from contaminating the components on route to or at the sampling site before assembly.

5.3.2 Use of the Sampling Trains

Figure 5-9 illustrates the sampling procedures used by each test crew at the different test locations. When a crew arrived at the sampling site, they hoisted or carried the sealed components to the sampling platform, assembled the sampling trains, and positioned them at a specific sampling port. Figure 5-10 illustrates the sampling train breakdown procedures used by each crew. Figure 5-11 shows the equipment arrangement at port C (Methods 5 and 8) and port A (Methods 3 and 4) on the main smelter stack. Figure 5-12 shows the equipment arrangement at port A at the reverb test site.

Preliminary checks and measurements were made as follows. While the particulate sampling train was used in one sample port, the combined gas composition and moisture train was used in another sample port, so that measurements were made simultaneously. The particulate sampling trains traversed the ducts or stacks, while the gas composition and moisture trains remained at a fixed sampling point.

In addition, the sampling crews leak checked the sampling trains before each test by fastening a blank swagelok to the end of the probe, drawing a 15-inch Hg vaccum on the systen, and recording the movement of the dry gas meter needle for 1 minute. If the leak rate was greater than 0.02 cfm, the source of the leak was found, corrected, and another leak check was performed.

The magnehelic gages for ΔP and ΔH were then zeroed and levelled and all data specified by EPA Methods 5 and 8 were recorded on standard data sheets, as shown in Figure 5-13.

After the equipment was readied, the process observer (Chiba) notified the supervisor of the three test crews (Steiner) of the status of the process. Since EPA Region IX specified that all tests must take place during the copper blow portion of the converter cycle, the process observer was stationed at the acid plant control room at the beginning of each test to inform the test supervisor of the start of copper blow. The supervisor then notified each test crew leader to begin sampling. Normally, all sampling trains were started within approximately 5 minutes after copper blow.

Figure 5-11. Equipment arrangement at main smelter stack, ports A and C.

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Figure 5-12. Equipment arrangement at reverb ESP outlet, port A.

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[illegible] PLANT
LOCATION
STACK DIAMETER (IN.)
DUCT DIMENSIONS (IN.) X (IN.)

4,01 DATE
TIME
RUN

4,02 OPERATORS
STATIC PRESSURE (IN.HG.)
4,03 AMBIENT PRESSURE (IN.HG.)
TYPE S PITOT COEFFICIENT

STACK PRESSURE (IN.HG.).
4,04 MOLECULAR WEIGHT (LB/LB-MOLE)
B_{wo}

METER BOX NUMBER
ORIFICE METER COEFFICIENT
4,05 PROBE LENGTH (FT.)
NOZZLE DIAMETER (IN.)

PARTICULATE SAMPLING DATA

4,06

Sampling Point Number	Clock Time	Stack Gas Temp (°F)	Probe Temp (°F)	Impinger Outlet Temp (°F)	Oven Temp (°F)	Dry Gas Meter Temp Inlet (°F)	Outlet (°F)	Velocity Head (in. wg.)	Orifice Meter (in. wg.)	Gas Meter Volume (ft.³)	Pump Vacuum (in. hg.)
		Average				Average	Average		Average	Total	
						Average					

EOF

Figure 5-13. Particulate sampling data sheet.

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During the test, the process observer toured the smelter to observe various parts of the operation (in particular, the reverb furnace and the acid plant) to insure that normal operating conditions prevailed. And at the same time, the test supervisor observed the sampling practices of one or more of the test crews to insure the tests were being conducted in accordance with Methods 5 and 8.

Figure 5-14 shows, as a function of time, how the simultaneous tests were generally carried out. Normal operation of the converter during a typical copper blow gave a total of 2 hours of available sampling time for each test. Nevertheless, there were variations in the actual time required to complete the different tests, due to the difficulty in moving the sampling train from one port to another (easy at the reverb, difficult at the main stack, very difficult at the acid plant) and the number of ports sampled. (See Volume III, Appendix A for rest of data.)

Simultaneous sampling was difficult to coordinate, as Phelps-Dodge personnel were not able to predict the start of a copper blow with any degree of consistency. Occasionally, the test crews would prepare the equipment to sample at a specific time (estimated by Phelps-Dodge personnel) and then the start time would be advanced or delayed. Since it was more difficult to prepare for a test at some sites than others, one crew was usually ready to start before another. Also, if a leak was detected during the leak check, it had to be corrected, creating another delay. Rather than jeopardize the entire test for a given copper blow, the crews started sampling when they were ready once they had been notified that a copper blow had started. In spite of this, careful coordination between the process observer, the supervisor, and the test crews kept the start times fairly uniform throughout the test program.

During a test, each sampling crew followed the same basic procedure:

- Allow the sampling train to heat up to 250°F
- Position the pitot tube and nozzle to face directly into the gas streamline lines in the duct or stack
- Seal the sampling probe in the sample port (except reverb)
- Measure the ΔP at the sampling point, calculate ΔH required for isokinetic sampling, and set this value on the orifice magnehelic gage (proportional sampling was used for gas and moisture)
- Record all the required data on the pertinent data sheet
- Maintain isokinetic conditions throughout the traverse.

After a traverse was completed, the sampling train was moved to the next sample port. If the sampling train was disassembled during the transfer, as at the main stack and acid plant, a leak check was made before starting the next traverse. The procedure described above was repeated for all tests.

At the conclusion of a test, the sampling train was removed from the duct or stack and was allowed to cool down. The impinger train was disconnected and purged for 10 minutes with ambient air to transfer any SO_2 retained in the 80 percent IPA into the H_2O_2 solution. When the components were cool enough to handle, the sampling train was disassembled at the test site piece by piece. As soon as a component was removed, its inlet and outlet were sealed by stretching parafilm over the openings to prevent contamination of the sample by fugitive dust at the sampling site or during transit to the lab. The various components (nozzle, probe, filter holder, impinger train from Methods 5 and 8, and Tedlar bag and impinger train samples from Methods 3 and 4) were then transported to the laboratory for sample recovery and analysis, as previously summarized in Figure 5-10.

Section 6 of the report details the sample recovery procedures used by Aerotherm personnel.

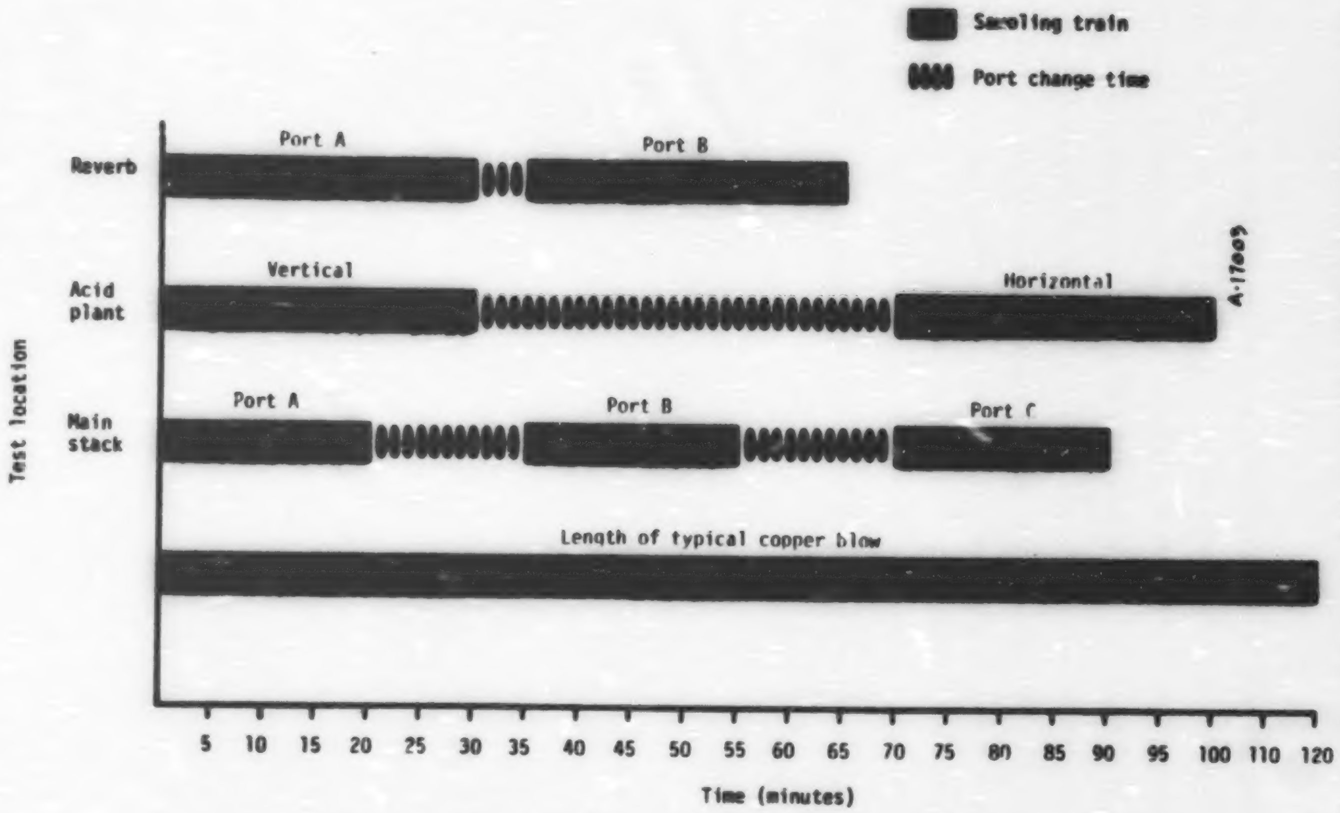

Figure 5-14. Sampling schedule for simultaneous stack tests.

SECTION 6

SAMPLE RECOVERY AND HANDLING PROCEDURES

This section describes the procedures employed to recover the samples from the three types of sampling trains used during the field test program (the combined Methods 5 and 8 train, the combined Methods 3 and 4 train, and the in-stack train). After completion of a test, the sealed components were transported back to the laboratory, where sample recovery could be performed in clean conditions to prevent any contamination of the samples. The following subsections describe the specific procedures used to recover samples from each type of train. Exceptions to these procedures may be found in Appendix A, Daily Activities Report.

6.1 EPA METHODS 5 AND 8 SAMPLE RECOVERY PROCEDURES

Particulate matter was recovered from the sampling nozzle, sampling probe, and filter holder of each sampling train. After removing the parafilm cover from the inlet and outlet openings of the sampling nozzle and probe, both components were rinsed with analytical reagent grade acetone, brushed with a nylon brush and rinsed again with acetone. This procedure was repeated at least twice until the rinse solution appeared clean to the naked eye. These acetone washings were put into a polyethylene bottle labeled as shown in Figure 6.1 with the following information: date of test, the test number, the type of sample, and the test location. Three sets of these labelled polyethylene containers were used to identify and separate the samples from the three test locations.

In some cases, the pyrex glass liner of the sampling probe was broken during testing. When this happened, only a partial sample was recovered from the probe because of the possibility of contamination by the glass liner itself. On several occasions, the nozzle from the acid plant had a large amount of particulate sludge just inside the nozzle tip. It is believed that the sampling nozzle scraped this sludge from the 20-inch long sample port while the probe was being removed from the duct. This particulate sludge was removed from the nozzle using a small spatula and was discarded before the nozzle was cleaned with acetone.

After particulate samples had been recovered from the sampling nozzle and probe, the filter holder was opened with the inlet side facing upwards. The 142 mm filter was then removed from the holder with a pair of tweezers and returned to the original labelled petri dish from which it had

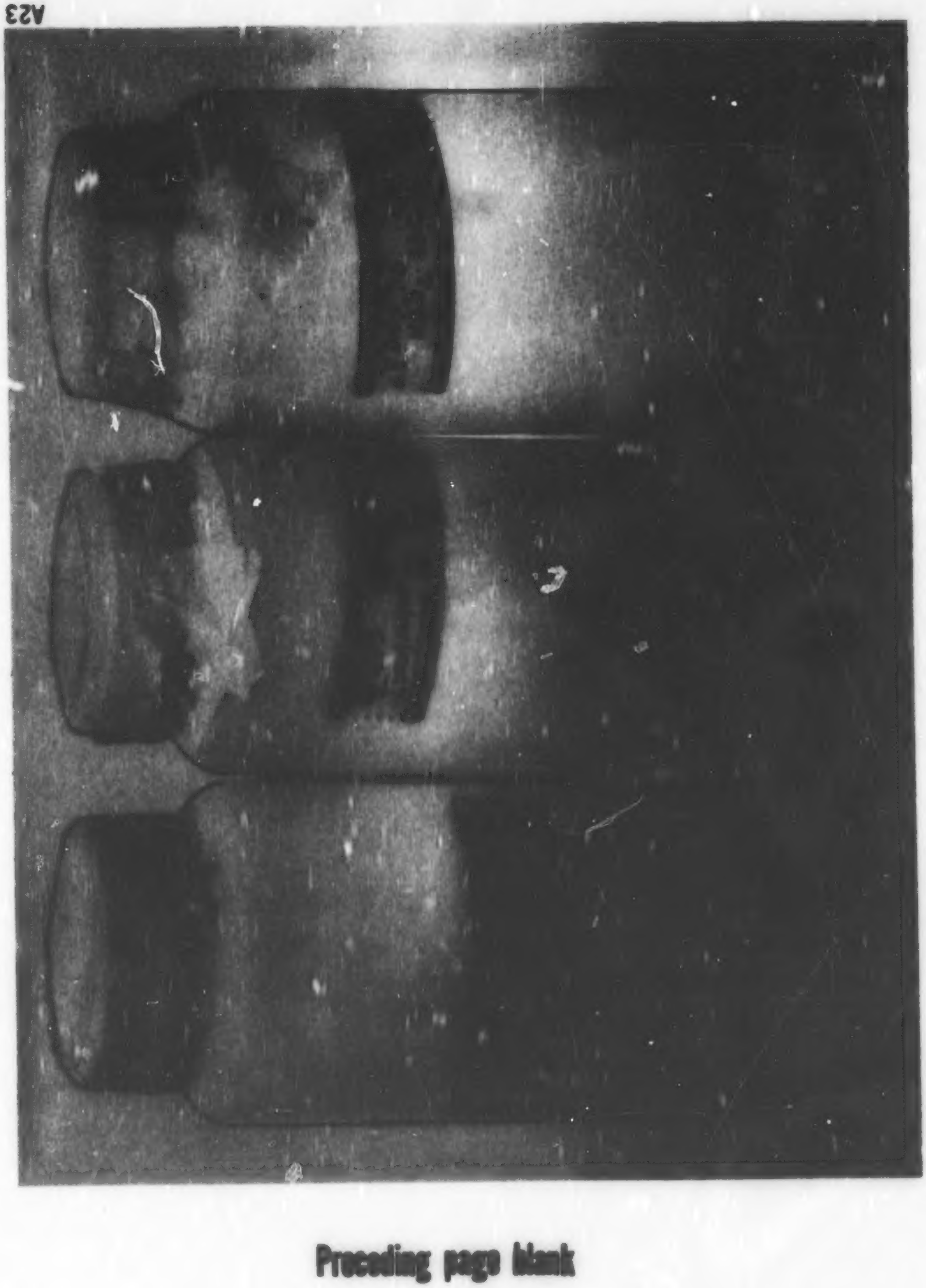

Figure 6-1. Polyethylene storage bottles.

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been taken, as shown in Figure 6.2. In some instances, fibers from the filter adhering to the surface of the filter holder had to be scraped off with the tweezers. In doing this, care was taken not to remove the teflon coating on the filter holder. The petri dish containing the used 142 mm filter was sealed with tape and labelled a second time with the date of the test, the test location, and the test number.

There was one exception to this basic sample recovery procedure. Apparently, the reverb furnace gas stream contained some unknown constituent which attacked the teflon coating on the internal surfaces of the filter holder and caused it to flake. As the stainless steel sealing surface was exposed, more and more filter fibers stuck to this surface. In these cases, extreme care was taken to remove the fibers from the holder without removing the teflon with the fibers. Frequently, fibers were left on the sealing surface because they could not be removed without being contaminated by flakes of teflon. This loss of fibers would tend to underestimate the particulate matter emission rate.

After the filter had been removed, the internal surfaces of the filter holder were rinsed with analytical reagent grade acetone and brushed with a nylon brush. (This step was omitted for the filter holders which showed evidence of teflon flaking to avoid contaminating the sample with teflon.) These acetone washings were added to the polyethylene bottle containing the probe and nozzle washings. The cap for the bottle was screwed on tightly and tape was wound around the cap to insure that the bottle did not leak. Labelled sample bottles and petri dishes were then set aside in numbered shipment boxes and the numbers recorded to identify which samples were shipped in which boxes.

After recovering the particulate sample from the front half of the sampling train, the test crew proceeded to recover the SO_3/H_2SO_4 and SO_2 samples from the back half of the train. The contents of impingers #1 and #2 were transferred to a polyethylene sample bottle. These impingers were then thoroughly rinsed with an 80 percent IPA solution and the washings put into the polyethylene bottle. The backup filter holder which contained the 50 mm glass-fiber filter was disassembled and the filter added directly to the polyethylene sample bottle. The front half of this glass filter holder was then rinsed with 80 percent IPA solution and these washings also added to the sample bottle. The top for this bottle was screwed on tightly and tape was wound around the cap. The information on the label of each sample bottle containing the IPA sample solutions is illustrated in Figure 6.1.

The same basic procedures were used to recover the H_2O_2 sample solutions in impingers #3, #4 and #5. The solutions from impingers #3 and #4 were transferred to a labelled polyethylene sample bottle along with any condensed liquid from impinger #5. The back half of the backup

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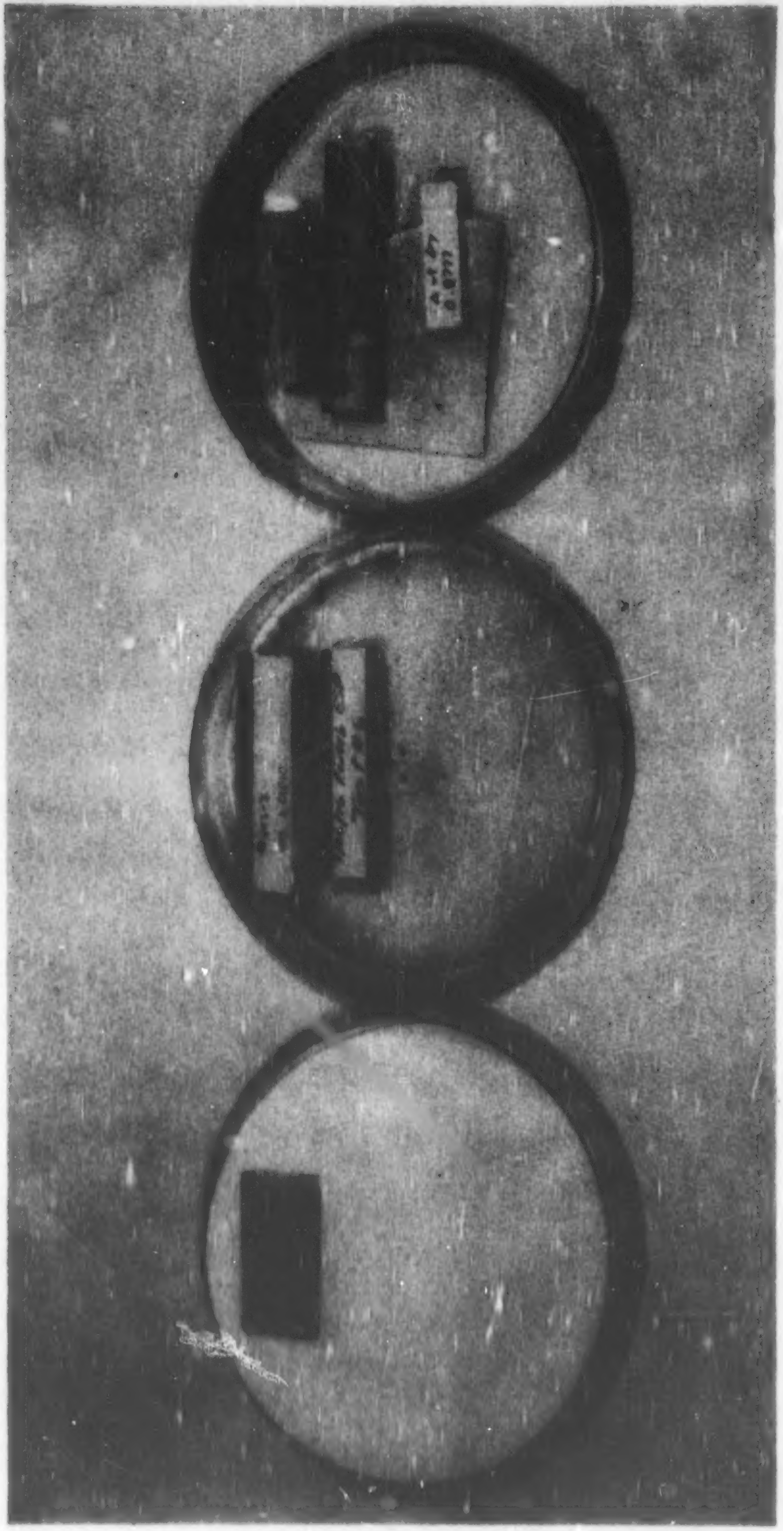

Figure 6-2. Petri dishes for 142 mm filters.

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filter holder and the internal surfaces of impingers #3, #4 and #5 were then thoroughly washed with distilled water, and the washings were added to the sample bottle. As before, the cap to the sample bottle was screwed on tightly and taped. The date of the test, the test number, the type of sample and the test location were all recorded on the label. Both the IPA and H_2O_2 sample bottles were then set aside in a numbered shipment box, and the information on the labels of these two bottles — as well as the shipment box number — was recorded.

6.2 EPA METHODS 3 AND 4 SAMPLE RECOVERY PROCEDURES

The integrated sample of the stack gas collected in the tedlar bag was taken to the laboratory for analysis with the Orsat analyzer. Since the sample was a gas, no sample recovery was necessary. The tedlar bag was simply connected directly to the Orsat analyzer and the gases pumped into the burette. The burette and the glass tubing of the Orsat were then purged three times with the gases in the tedlar bag to condition the internal surfaces of the Orsat. A measured volume (100 ml) of the gases was then drawn into the burette and sequentially passed through three fresh absorption solutions which removed CO_2, O_2, and CO in that order. Several passes were made through each solution to insure quantitative capture of each gaseous species. The results of this analysis were recorded on the appropriate field data sheet, and the tedlar bags were pumped out and made ready for use on the next test.

Since no chemical analysis of the moisture train samples was required, recovery of the moisture samples from the combined Method 3 and 4 sampling trains at the reverb and main stack sampling sites took place in the field. (No moisture measurements were made on the effluent from the acid plant.) At the completion of a test, the moisture sampling train was disassembled. The flexible teflon hose which connected the probe and filter to the impinger train (immersed in the icebath) was then drained into a labelled polyethylene sample bottle to insure that no condensed water remained in the hose. Each of the Lexan impingers containing condensed water were also emptied into the same sample bottle. The cap for the bottle was screwed on tightly and the bottle was set aside for transportation to the lab. Separate sample bottles — the same ones that were used to transport distilled water and fresh silica gel to the sample site prior to the test — were used to store the used distilled water and the spent silica gel.

After the sealed sample bottles had been transported to the laboratory, the volume of water in the first sample bottle was measured with a graduated cylinder and recorded on the stack gas moisture data sheet. The weight of the spent silica gel in the second sample bottle was measured on a triple beam balance and also recorded on the data sheet. The water and silica gel were then

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discarded. The sample bottles were cleaned out, dried, and charged with measured amounts of distilled water and silica gel for the next test.

The next subsection describes the sample handling procedures for the recovered samples.

6.3 EPA METHODS 5 AND 8 SAMPLE HANDLING PROCEDURES

After the samples had been recovered in the laboratory, they were put into sealed, labelled containers as previously described and transported to the motel in a numbered shipment box. Once a sufficient number of samples had been collected to fill a shipment box, the box was closed, sealed with tape, and a mailing label put on the outside of the numbered box. Rather than ship each box separately and risk loss or damage each time, the shipment boxes were stored in the motel until a total of nine boxes had been accumulated. These nine boxes were then shipped by the shipping and receiving department of Phelps-Dodge. A local carrier (Milne) trucked the shipment to Phoenix, where it was transferred to an interstate carrier (Leeway) for shipment to Aerotherm in Mountain View, California.

The sample shipment was received in good order by Aerotherm's shipping and receiving department and transferred to the Source Evaluation Laboratory shown in Figure 6.3. The samples were stored in this locked laboratory (restricted to authorized personnel) for chemical analysis. When inspected by the analyst, none of the boxes appeared damaged and all were still sealed. Examination of the contents of each box showed that all sample bottles were still sealed with tape and were undamaged.

This first shipment included samples from test #1 through #7. The remaining samples (tests #8 through #12), were boxed as described above and were taken to Hayden, Arizona to be shipped along with the samples to be obtained at the Kennecott Smelter. In the meantime, this second set of samples were stored in the mobile equipment van which was locked at all times. At the completion of the Kennecott field test program, the test crew delivered these samples to the air cargo terminal at the Phoenix Airport, where the samples were transferred to an interstate carrier (Leeway) and delivered to Aerotherm. As before, the samples were received in good condition, with no signs of damage or entry.

Throughout the analysis program, all samples were kept in their sample containers and stored in the analytical balance room or the Source Evaluation Laboratory. Both of these facilities are locked and admittance is restricted to authorized personnel only.

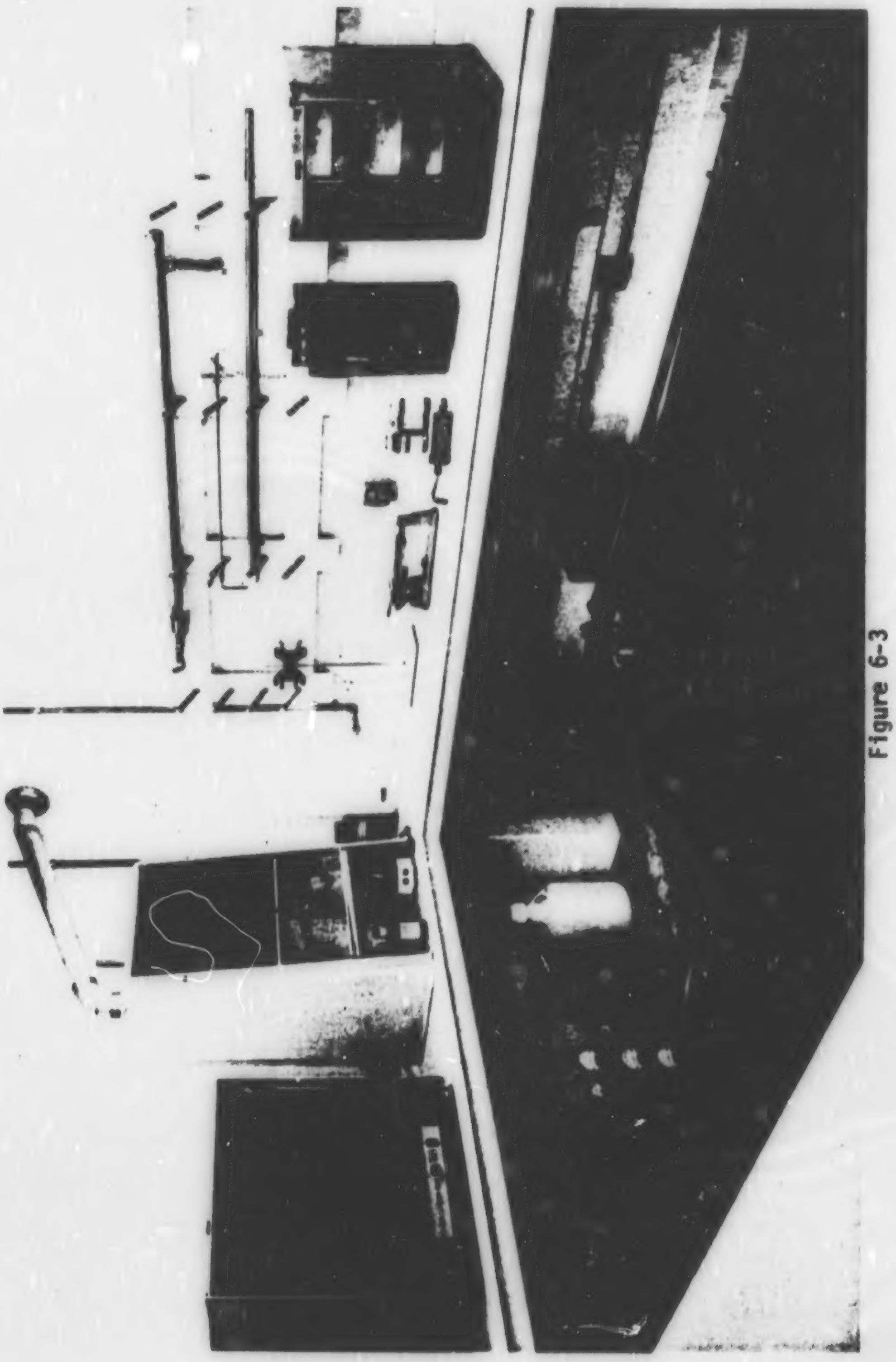

Figure 6-3

Emissions Source Evaluation Laboratory

Since the completion of the analysis program, all samples are now being stored in sealed sample containers in Aerotherm's Source Evaluation Laboratory. These samples can be retained for evidence and analyzed at a later date to determine their chemical composition. In addition, used filters from the combined Method 3 and 4 sampling trains are also being stored and can be analyzed immediately, if desired, since they are not required for evidence.

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SECTION 7

PHYSICAL AND CHEMICAL ANALYSIS PROCEDURES

This section describes the procedures used to weigh the filters, evaporate the acetone washings and weigh the residue, and analyze the impinger solutions for SO_3/H_2SO_4 and SO_2.

7.1 WEIGHING THE PARTICULATE FILTERS

After the completion of each test, all the filters used in the combined Method 5 and 8 sampling trains were sealed in petri dishes for shipment from Arizona to California. During this shipment some of the particulate on the surface of the glass fiber filters was dislodged into the petri dish. Hence, two samples had to be recovered — one being the filter and the other being the loose particulate. Sample recovery took place in Aerotherm's Source Evaluation Laboratory.

To recover the loose particulate in the petri dish, the lid of the dish was first rinsed with a small amount of distilled water to remove any particulate adhering to the surface and then wiped dry. Next, the glass fiber filter was removed from the petri dish, and the bottom dish was rinsed with distilled water and wiped clean. The washings from the petri dish were added to the polyethylene sample bottle containing the acetone washings from the probe, nozzle, and filter holder for that particular test.

After the loose particulate in the petri dish had been recovered, the glass fiber filter was transferred to the bottom half of the petri dish. The dish was then put into a desiccator containing silica gel and desiccated for at least 24 hours. After desiccation was complete, the filter and petri dish were transferred to a small, portable desiccator. They were then taken to the balance room to be weighed on a Mettler Model H20 analytical balance which had been levelled and zeroed. The 142 mm filter paper was removed from the desiccator with tweezers and folded in half twice so that it would fit on the balance pan. Because of the hygroscopic nature of the particulate matter on the filter, the filter was gaining weight very rapidly by adsorbing water vapor from the air. As a result, no attempt was made to determine the exact weight of the filter at that point. Instead, an approximate weight was determined as rapidly as possible, and the filter was returned to its petri dish and was desiccated for another 24 hours.

After being desiccated a second time, the filter was ready to be weighed accurately. When the approximate weight of the filter had been set on the balance, the filter was placed on the balance pan. The analyst immediately started a stopwatch, and the weight of the filter was measured and recorded 10 to 20 seconds after the filter was placed on the pan. As each filter gained weight by adsorbing water vapor, the analyst continued to take readings until the filter reached equilibrium - up to 24 hours for some filters.

After completing the weight versus time curve (see Volume III Appendix B for example), the analyst returned the filter to its petri dish, labelled the dish with a gross weight, and returned to sample to the Source Evaluation Lab for storage pending further instructions from EPA Region IX.

7.2 NOZZLE, PROBE AND FILTER HOLDER ACETONE WASHES

The polyethylene sample bottle containing the acetone washings from the sampling nozzle, the sampling probe, and the filter holder — as well as the distilled water washings from the petri dish — was opened and placed in the fume hood shown in Figure 6.3. The sliding window of the hood was partially closed and the fume hood fan turned on to circulate laboratory air through the hood. After the acetone in the sample bottle had evaporated until approximately 5 ml of acetone remained in the bottle, the slurry was transferred to a tared beaker (usually 10 ml or 30 ml beaker with a small,tare weight) and evaporated to dryness in the fume hood. Because of the presence of water in the original sample (petri dish washings), many of the samples did not evaporate to total dryness. In these cases, the beakers were placed on a steam bath and evaporated to dryness. Figure 7.1 illustrates the residue that remained in the beaker after evaporation.

After evaporation, each beaker was transferred to the desiccator and desiccated for 24 hours. The tared beaker was then transferred to the portable desiccator and taken to the balance room. Unlike the particulates on the glass fiber filters, the residues were not hygroscopic and could be weighed directly. After the weight of each sample was recorded in the filter record book, the samples were stored in the Source Evaluation Laboratory pending further instructions from EPA Region IX.

7.3 SO_3/H_2SO_4 AND SO_2 SAMPLES

Before titrating the isopropanol solutions containing SO_3/H_2SO_4, certain standardizations were carried out in the laboratory. The analyst (Atkins) used a 1 N NaOH Acculute Standard volumetric solution from Anachemia Chemicals Limited to prepare a 0.01 N NaOH standardization solution

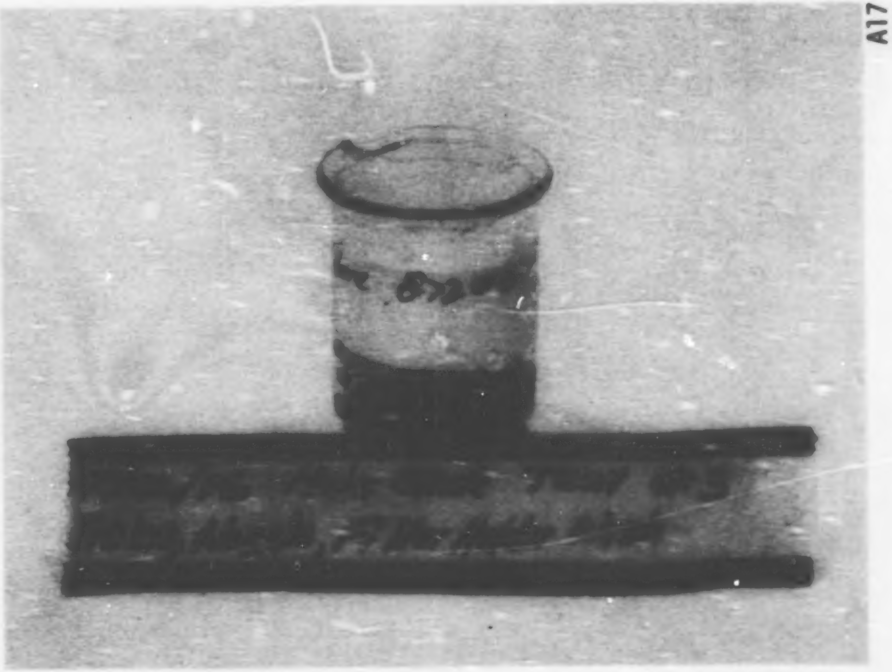

Figure 7-1. Residue after evaporation of acetone washings.

by dilution with deionized water. Several titrations using this NaOH standardization solution were used to repeatedly standardize the analytical reagent grade 0.01 N H_2SO_4 from Baker Chemicals. After the sulfuric acid solution had been standardized against the standard sodium hydroxide solution, the 0.01 N H_2SO_4 solution was in turn used to standardize the barium perchlorate solution. As before, this standardization check was done repeatedly and the average of several titrations was used to calculate the average normality of the $Ba(ClO_4)_2$ solution.

As a further check on the accuracy of the standard NaOH solution, it was standardized against a primary standard potassium acid phthalate solution from Baker Chemicals. The $KHC_8H_4O_4$ was dried in an oven at 105°C - 110°C for 1 hour and then allowed to cool in a desiccator. An accurately weighed amount was then added to a flask and dissolved in boiling deionized water. The solution was allowed to cool to room temperature and phenolphthalein indicator was added. When this primary standard solution was used to verify the accuracy of the standard NaOH solution, the maximum deviation between the two solutions was 0.0002.

The 50 ml buret used for all titrations was also calibrated. The buret was filled with water and a measured volume of water was delivered into a tared container. The container was then stoppered and weighed on the Mettler balance. The ambient temperature was recorded to convert the mass of water to volume, using data from Introductory Qualitative Chemistry by Olson, Koch, and Pimental. Since the difference between the delivered volume and the calculated volume was less than 1 percent in all cases, no correction factor for the buret reading was necessary. The same procedure was used to calibrate the pipets used in the titrations. Again, no correction factors were required. After performing all these standardization and calibration checks, the analyst was now prepared to perform the titrations of field samples.

7.3.1 Titration Procedures for SO_3/H_2SO_4 Samples

After the sample bottles containing the isopropanol solutions were shaken to make sure the solution was homogeneous, a measured aliquot (varying from 1 ml to 100 ml) was transferred to a flask with a calibrated pipet. Two to four drops of thorin indicator were then added and the aliquot was titrated with the standardized barium perchlorate solution to a pink end-point. In addition, a blank (isopropanol and deionized water) of approximately the same size of aliquot was titrated in a similar manner. The following data was recorded in the analytical log book:

- The volume of barium perchlorate titrant used to titrate the aliquot
- The volume of barium perchlorate titrant used to titrate the blank

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- The normality of the barium perchlorate titrant
- The total solution volume of sulfuric acid (first 2 impingers and filter)
- The volume of the aliquot titrated.

Two aliquots from each sample container were titrated.

7.3.2 Titration Procedures for SO_2 Samples

After the sample bottles containing the hydrogen peroxide solutions were shaken to make sure the solution was homogeneous, a measured aliquot (varying from 1 ml to 25 ml) was transferred to a flask with a calibrated pipet. A total of 100 ml of isopropanol and 2 to 4 drops of thorin indicator were added to the aliquot, which was then titrated to a pink endpoint using the standardized barium perchlorate solution. The following data were recorded in the analytical log book:

- The volume of barium perchlorate titrant used to titrate the aliquot
- The normality of the barium perchlorate titrant
- The total solution volume of sulfur dioxide (third and fourth impingers)
- The volume of the aliquot titrated.

These titration data were used to calculate the concentrations of SO_3/H_2SO_4 and SO_2 in the various gas streams. The results of these calculations are presented in the following section.

SECTION 8

RESULTS AND CONCLUSIONS

This section of the report summarizes the results of the field tests and draws conclusions after analyzing these results.

8.1 CALCULATING PROCESS WEIGHTS AND ALLOWABLE EMISSION RATES

In order to determine whether or not the three stationary sources sampled at the Phelps-Dodge smelter were in compliance with the applicable particulate emission regulations, the process weight at each source had to be determined. Separate process weights were calculated for the acid plant (using converter process weight data) and the reverb furnace. The sum of these process weights was then used to determine the allowable emission rate from the main stack of the Ajo smelter. A description of the procedures used to compute these process weights follows.

8.1.1 Process Weight and Allowable Emission Rate for the Acid Plant

There were only two converters (#1 and #3) in operation on the days the stack sampling tests were performed. When one converter was blowing copper, the other converter was idle and the damper above that converter was closed. Hence, only the weight charged into the one converter blowing copper during a test was considered.

The total process weights for the acid plant were calculated directly from the daily converter logs supplied by the Phelps-Dodge staff. The mass inputs shown on these logs (ladles of matte, bins of flux, etc.) were converted to tons by using appropriate conversion factors (see sample calculation). The material introduced into the converters included matte (from the reverb), flux, chips, and dust. Sometimes concentrate was also added to the converter, but the quantity involved was small and was neglected.

To figure the weight of material introduced into the converter blowing copper during stack sampling tests, the total process weight introduced over a 24-hour period was first calculated.

A 24-hour period was chosen because the converter process is a batch type operation, which means that the mass inputs must be averaged over a reasonably long period of time to obtain representative values. During this 24-hour period, the intervals during which matte and copper blows took place (as shown in the daily converter log) were summed to obtain the total process time. The emissions from the converter were significant only when air was being blown into the converter. During the rest of the time the converter was assumed to be idle, since it was either empty, loading or unloading, or simply holding copper.

The process weight rate for the converters was then calculated by dividing the process weight introduced in a particular 24-hour period by the process time (i.e., the sum of all matte and copper blow times) during that period.

This procedure for calculating process weight rate is demonstrated here with a sample calculation. On July 26, 1976, stack sampling tests were run during the copper blow on Converter #1 between 8:50 and 11:20 a.m. Figure 8-1 is a copy of the daily converter log for Converter #1 on that day. The first three columns of the log show the material introduced into the converter; in the next two columns, the start and stop times of copper and matte blows were recorded.

The log shows that a total of 12 ladles of matte were introduced into the converter that day. Besides these ladles of matte, 10.25 flux bins and four revert (i.e., chips) boats were added. A collar was pulled once that day, and no boats of dust were added. (Revert boats usually contain some dust with the chips, but if a boat contains dust alone, or mainly dust, this is indicated in the reverts boats column.) The conversion factors to change these units into tons were estimated by the Phelps-Dodge staff to be approximately as follows:

- 1 ladle of matte = 15.75 tons
- 1 bin of flux = 3 tons
- 1 boat of chips = 8 tons
- 1 boat of dust = 4 tons

In addition, it is estimated that a collar pull adds 4 tons of chips to the charge. Using these conversion factors, we can calculate that 189 tons of matte, 30.75 tons of flux, and 36 tons of chips and dust were introduced into the converter on July 26, 1976. This means that the total process weight in the 24-hour period was approximately 256 tons. (Note that no transfer of matte from one converter to another is indicated on the log. If matte is transferred from a converter to the converter under consideration, then the number of ladles transferred must be included in the mass input.)

The intervals during which air was blown into the converters is indicated in Columns 4 and 5 on Figure 8-1. The A shift begins at 7:00 a.m., and if the converter is blowing at the beginning of the shift, the blowing is considered to begin at 7:00 a.m. for the following 24-hour period. For

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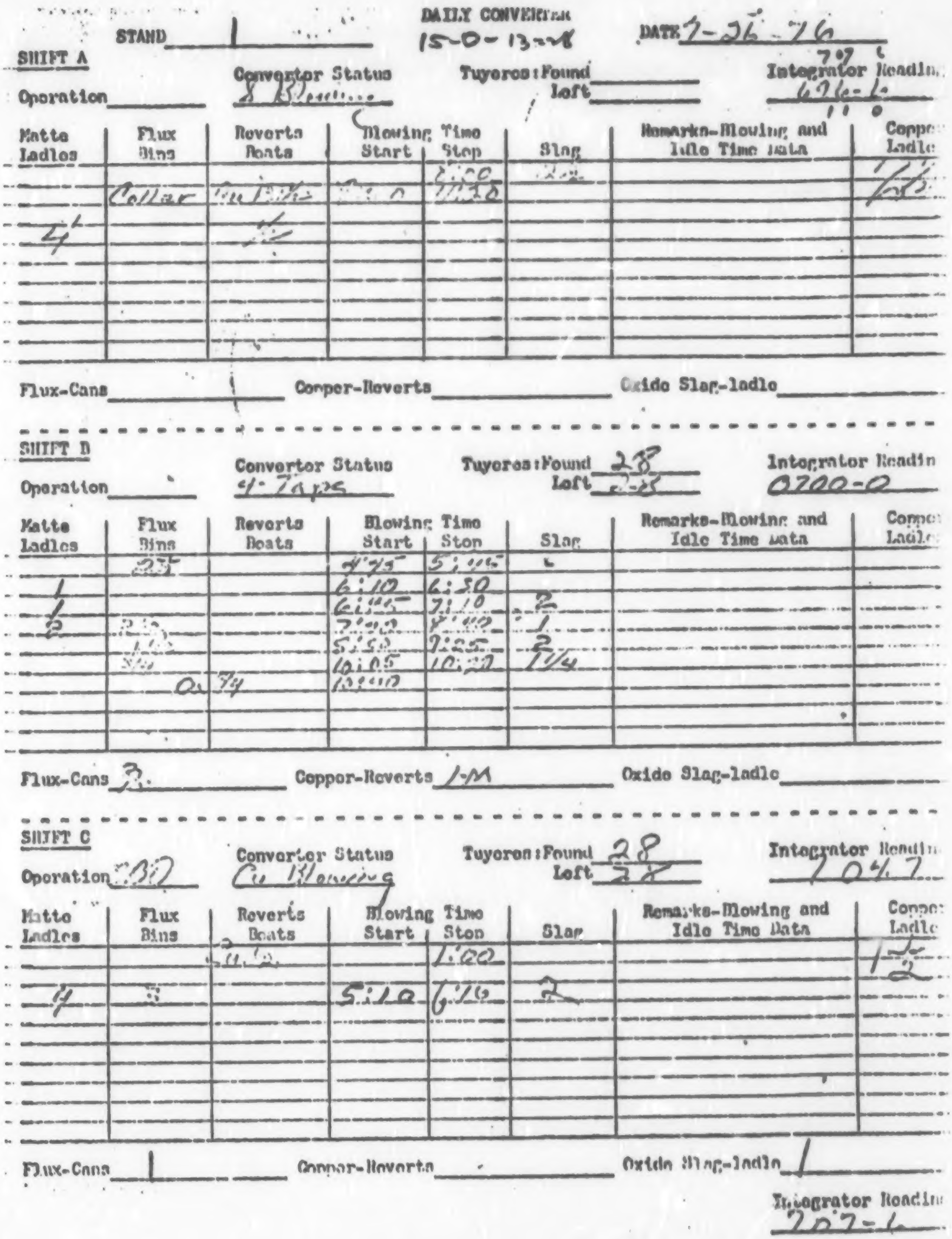


DAILY CONVERTER
15-0-13-28

STAND 1

DATE 7-26-76

SHIFT A

Operation ______

Convertor Status 8 Blowing

Tuyeres: Found ______
Left ______

Integrator Reading 707 / 676-6

Matte Ladles	Flux Bins	Reverts Boats	Blowing Time Start	Blowing Time Stop	Slag	Remarks-Blowing and Idle Time Data	Copper Ladle
				2:00	[illegible]		1/2
	Collar	[illegible]	[illegible]	11:50			[illegible]
4		4					

Flux-Cans 1

Copper-Reverts ______

Oxide Slag-ladle ______

SHIFT B

Operation ______

Convertor Status 4-Taps

Tuyeres: Found 28
Left 28

Integrator Reading 0700-0

Matte Ladles	Flux Bins	Reverts Boats	Blowing Time Start	Blowing Time Stop	Slag	Remarks-Blowing and Idle Time Data	Copper Ladle
	25		4:45	5:05			
1			6:10	6:30			
1			6:45	7:10	2		
2	[illegible]		7:00	8:00	1		
	[illegible]		8:50	9:25	2		
	[illegible]		10:05	10:20	1 1/4		
	Cu	74	12:00				

Flux-Cans 3.

Copper-Reverts 1-M

Oxide Slag-ladle ______

SHIFT C

Operation 307

Converter Status Cu Blowing

Tuyeres: Found 28
Left 28

Integrator Reading 1047

Matte Ladles	Flux Bins	Reverts Boats	Blowing Time Start	Blowing Time Stop	Slag	Remarks-Blowing and Idle Time Data	Copper Ladle
		[illegible]		1:00			1 1/2
4	[illegible]		5:10	6:16	2		

Flux-Cans 1

Copper-Reverts ______

Oxide Slag-ladle 1

Integrator Reading 707-6

Figure 8-1. Converter log for July 26, 1976.

July 26, 1976, the log shows that the total blowing time was 10 hours and 25 minutes. Although we are not concerned here with differentiating between matte blow and copper blow intervals, the copper blow times are clearly marked on the log by the designation "Cu" to the left of the copper blow intervals. All the rest are matte blows.

The formulas used to calculate the allowable particulate emission rates for processes of this type are taken from CFR Title 40 Subsection 52.126. These formulas are:

$$E = 3.59\ P^{0.62} \quad \text{where} \quad P \leq 30 \text{ tons/hr}$$

$$E = 17.31\ P^{0.16} \quad \text{where} \quad P > 30 \text{ tons/hr}$$

where E = emissions in pounds per hour

P = process weight in tons per hour

Using the figures from the log for July 26, 1976, the process weight rate for the acid plant converter was 256 tons divided by 10 hours 25 minutes, or 24.6 tons/hour. Since P is less than 30 tons/hour, we use the first equation to figure a maximum allowable particulate emission rate from the converter of 26.1 lb/hour.

8.1.2 Process Weight and Allowable Emission Rate for the Reverb Furnace

The total solid mass introduced into the reverberatory furnace is calculated over an 8-hour shift period during which a stack sampling test was made. The solid materials introduced into the reverb include concentrate, converter slag, precipitate, lime, and dust. The weights of these materials charged to the furnace during the sampling tests were supplied by the Phelps-Dodge staff at Ajo, Arizona. The sum of the weights represents the total process weight. Since a reverb furnace operates continuously, the total process weight divided by the hours in a shift gives the required process weight rate.

The total solid input into the reverb furnace during Shift A on July 26, 1976 was estimated by the Phelps-Dodge staff (see Figure 8-2) to be as follows:

Concentrate = 175 tons

Converter slag = 116 tons

Precipitate = 6 tons

Lime = 15 tons

Dust = 8 tons

1 Converter Gas Stream.

Copper Blow Test on A shift of 7/2:

	Taps	Matte Tons	Flux Tons	Chip Tons	Copper Blow Min	Cu Tons
Converter No 1	8	126	23	20	150	52
Converter No. 2	-					
Converter No 3.						

②

Total Weight Charged to Converter on A shift of 7/26

	Matte in Ton.	Chips	Flux	Conc.	Total
Converter No. 1	63	20	0	0	83
Converter No 2					
Converter No 3					

Total Input into Reverb Furnace. on A shift of 7/26

Natural Gas	Air	Conc.	Conv. Slag	P.P.	Lime	Dust
176,000 cft/hr.	180,000 CFM.	175	.116	6	15	8

Figure 8-2. Reverb furnace process data.

By adding up these weights, we get a total solid input for the 8-hour period encompassing the test of 320 tons. As the reverb operation is continuous, the process weight is simply 320 divided by 8, or 40 tons/hour. As P is greater than 30 tons/hour, we can use the second equation in Section 8.1.1 to figure a maximum allowable emission rate from the reverb of 31.2 lb/hour.

8.1.3 Process Weight and Allowable Emission Rate for the Main Stack

The effluent from the acid plant and reverb furnace combine and enter the base of the main stack of the smelter. In addition, particulates and gas are delivered to the base of the stack by the fugitive gas collection system in use on the converters and the reverb furnace. When the DMA plant is operating, a portion of the reverb furnace gas is also sent to this plant, which has its own stack. But since this plant was not operational during the field test program, all the reverb furnace gas was sent to the main stack.

To obtain the process weights and allowable emission rates for the main stack (i.e., the smelter itself) the values calculated for the acid plant and reverb furnace were simply added to get a maximum allowable emission rate of 57.3 lb/hour.

8.2 RESULTS OF THE FIELD TESTS

Table 8-1 summarizes the sampling data from the field test program. Table 8-2 shows the emission rates for SO_2 and SO_3/H_2SO_4, and compares the measured particulate emission rates and the allowable particulate emission rates for each test location. Table 8-3 summarizes the actual sampling times.

The measured particulate emission rates for the reverb furnace and the main stack of the Ajo smelter exceed the allowable particulate emission rates by a substantial amount.

8.3 DISCUSSION OF FIELD TEST RESULTS

This subsection discusses the results of the field tests at each of the three sampling locations.

8.3.1 Acid Plant Test Results

An examination of the emission data in Table 8-2 shows that all tests conducted on the effluent from the acid plant were within the ±10 percent isokinetic sampling guideline required by EPA Method 5. As a result, no inaccuracies in emission data were introduced by a failure to meet these guidelines.

TABLE 8-1. SUMMARY OF SAMPLING DATA

T-29

Location	Run #	Date	Bwo x 100 (% moisture)	Vm, std. (scf)	CO_2 (%)	O_2 (%)	Md (lb/lb-mole)	Ms (lb/lb-mole)	Vs (fps)	Qs (scfh x 10^6)
Ajo Mainstack	1	7-20-76	2.5	29.4	1.2	17.7	28.90	28.63	16.1	14.0
	2	7-21/76	1.6	45.0	1.6	17.6	28.96	28.78	15.8	14.0
	3	7-22-76	2.5	49.2	1.4	16.7	28.90	28.62	21.0	18.5
	4	7-23-76	2.3	45.7	1.9	17.1	28.99	28.69	17.4	15.0
	5	7-26-76	6.2	38.3	1.7	17.3	28.96	28.28	14.3	11.9
	6	7-26-76	6.5	34.4	1.7	16.9	28.94	28.23	12.9	10.8
	7	7-27-76	6.7	26.8	1.6	17.3	28.95	28.22	9.4	7.8
	8	7-28-76	7.3	28.7	2.9	14.4	29.04	28.23	11.2	8.0
	9	7-28-76	5.3	38.2	3.0	13.4	29.02	28.43	15.5	11.3
	10	7-29-76	5.4	36.3	1.5	17.2	28.93	28.33	13.6	11.8
	11	7-29-76	4.3	30.0	1.5	17.4	28.94	28.46	11.4	10.0
	12	7-30-76	10.2	41.7	1.3	17.2	28.90	27.79	16.3	13.6
Ajo Reverb Esp	1	7-20-76	16.8	36.5	1.0	18.1	29.16	27.29	76.0	3.1
	2	7-21-76	12.3	44.8	1.0	18.1	28.88	27.54	81.4	3.6
	3	7-22-76	12.3	41.3	3.7	11.3	29.05	28.03	67.9	3.0
	4	7-23-76	12.3	44.2	0.9	18.1	28.86	27.53	84.3	3.8
	5	7-26-76	12.8	39.9	4.2	14.0	29.23	27.80	61.8	2.8
	6	7-26-76	10.1	44.7	4.2	14.0	29.23	28.10	77.4	3.6
	7	7-27-76	12.3	—	4.2	14.0	—	—	—	—
	8	7-28-76	9.0	49.9	5.9	11.3	29.39	28.36	90.1	4.2
	9	7-28-76	11.1	49.9	6.2	10.9	29.43	28.16	83.3	3.8
	10	7-29-76	12.1	45.2	6.3	10.5	29.43	28.05	77.5	3.9
	11	7-29-76	11.5	45.3	5.8	11.5	29.39	28.08	77.1	3.9
	12	7-30-76	13.8	45.2	5.8	11.5	29.39	27.83	72.1	3.6
Ajo Acid Plant	1	7-20-76	Dry	56.4	0.1	11.7	28.49	28.49	53.8	2.1
	2	7-21-76	Dry	55.1	0.1	11.7	28.49	28.49	53.1	2.0
	3	7-22-76	Dry	56.4	0.1	15.7	28.64	28.64	54.6	2.9
	4	7-23-76	Dry	55.9	0.1	15.7	28.64	28.64	54.2	2.0
	5	7-26-76	Dry	47.4	0.2	13.5	28.57	28.57	46.6	1.8
	6	7-26-76	Dry	65.5	0.4	11.0	28.52	28.52	58.1	2.3
	7	7-27-76	Dry	56.9	0.2	13.3	28.56	28.56	54.9	2.1
	8	7-28-76	Dry	53.9	0.1	13.9	28.57	28.57	54.5	2.1
	9	7-28-76	Dry	54.8	0.2	12.8	28.53	28.53	53.9	2.1

TABLE 8-2. SUMMARY OF PARTICULATE, SO_3/H_2SO_4 AND SO_2 EMISSION DATA

Location	Run #	Date	Particulate Concentration (lb/ft^3 x 10^{-6})			Particulate Emission Rate (lb/hr)			Percent Isokinetic (%)	Allowable Emission Rate (lb/hr)	SO_2 Concentration (lb/ft^3 x 10^{-6}), ppm	SO_2 Emission Rate (lb/hr x 10^3)	SO_3/H_2SO_4 Concentration (lb/ft^3 x 10^{-6}), ppm	SO_3/H_2SO_4 Emission Rate (lb/hr)
			Extrapolated Weight	First Measured Weight	Equilibrium Weight	Extrapolated Weight	First Measured Weight	Equilibrium Weight						
Ajo Mainstack	1	7-20-76	40.7 (0.2051)[a]	41.0 (0.287)[a]	58.9 (0.412)[a]	570.1	574.6	825.3	105.5	63.2	3.9 (2,360)[b]	5.5	1.6 (7.7)[b]	22.0
	2	7-21-76	39.6 (0.277)	39.7 (0.278)	54.8 (0.384)	553.3	554.8	766.2	106.9	63.9	4.6 (2,780)	6.4	0.1 (0.5)	1.6
	3	7-22-76	41.9 (0.293)	41.9 (0.293)	60.4 (0.423)	775.4	777.0	1118.2	87.8	62.6	4.4 (2,660)	8.2	0.3 (1.4)	6.4
	4	7-23-76	40.4 (0.283)	40.2 (0.284)	56.6 (0.389)	605.6	608.1	832.8	83.7	61.9	4.2 (2,540)	6.3	1.5 (7.2)	22.4
	5	7-26-76	38.6 (0.270)	38.8 (0.272)	54.5 (0.382)	458.7	461.3	647.0	104.8	57.4	7.1 (4,290)	8.4	0.9 (4.3)	10.8
	6	7-26-76	46.9 (0.328)	47.1 (0.330)	65.6 (0.459)	504.7	506.8	706.0	94.8	59.2	0.1 (60)	0.1	8.7 (42.0)	94.1
	7	7-27-76	42.7 (0.299)	43.3 (0.303)	66.9 (0.468)	333.7	338.3	522.6	115.2	61.3	5.4 (3,260)	4.2	8.5 (26.6)	13.3
	8	7-28-76	22.0 (0.154)	22.2 (0.155)	26.0 (0.182)	175.9	177.3	207.7	120.6	61.3	3.8 (2,300)	3.0	0.0 (0.0)	0.0
	9	7-28-76	75.1 (0.526)	75.2 (0.526)	111.9 (0.783)	847.7	849.5	1262.8	110.6	62.3	6.0 (3,620)	0.7	0.8 (3.9)	8.5
	10	7-29-76	44.2 (0.309)	44.5 (0.312)	67.1 (0.470)	520.0	524.5	790.3	99.6	62.4	5.0 (3,020)	5.9	1.4 (6.8)	16.8
	11	7-29-76	51.3 (0.359)	54.0 (0.378)	71.1 (0.498)	513.9	541.2	712.7	100.4	61.9	3.9 (2,360)	3.9	0.8 (3.9)	8.1
	12	7-30-76	43.2 (0.302)	43.6 (0.305	63.2 (0.442)	588.9	594.8	861.3	102.1	62.0	4.5 (2,720)	5.5	0.5 (2.4)	6.3
Ajo Reverb Esp	1	7-20-76	104.4 (0.731)	104.5 (0.732)	133.4 (0.934)	328.3	328.6	419.5	87.0	31.3	16.0 (9,660)	5.0	3.4 (16.4)	10.5
	2	7-21-76	88.1 (0.617)	88.2 (0.617)	107.4 (0.752	318.2	318.4	388.1	91.4	31.4	26.2 (15,820)	9.5	10.3 (49.7)	37.2
	3	7-22-76	91.3 (0.639)	91.4 (0.640)	114.4 (0.801)	272.9	273.2	341.9	109.2	31.6	11.2 (6,760)	3.3	5.6 (27.0)	16.8
	4	7-23-76	57.1 (0.400)	57.2 (0.400)	87.4 (0.612)	216.2	216.5	330.9	92.0	31.9	10.5 (6,340)	4.0	0.6 (2.9)	2.3
	5	7-26-76	63.4 (0.444)	63.4 (0.444)	97.0 (0.679)	175.3	175.6	268.4	107.7	31.2	8.0 (4,830)	2.2	0.0 (0.0)	0.0
	6	7-26-76	92.8 (0.650)	93.0 (0.651)	121.6 (0.851)	330.3	331.1	432.8	98.8	30.5	26.8 (16,190)	9.5	6.8 (32.8)	24.0
	7	7-27-76	-- --	-- --	-- --	--	--	--	--	30.8	-- --	--	-- --	--
	8	7-28-76	79.3 (0.555)	79.3 (0.555)	93.2 (0.652)	331.3	331.7	389.9	96.3	30.7	9.0 (5,440)	3.8	0.0 (0.0)	0.0
	9	7-28-76	71.5 (0.500)	76.9 (0.538)	132.3 (0.716)	273.3	293.9	390.9	104.0	31.7	3.0 (1,810)	1.1	8.8 (42.5)	33.7
	10	7-29-76	108.3 (0.758)	108.5 (0.760)	140.0 (0.980)	423.0	423.8	683.8	92.8	31.5	12.3 (7.430)	4.8	8.5 (41.1)	33.6
	11	7-29-76	111.0 (0.777)	111.5 (0.780)	135.8 (0.951)	431.8	433.6	528.2	89.7	31.0	6.3 (3,810)	2.4	8.4 (40.6)	22.7
	12	7-30-76	116.6 (0.816)	118.4 (0.829)	143.5 (1.004)	423.9	430.4	521.6	98.1	31.6	13.8 (8,340)	5.0	9.3 (44.9)	33.9
Ajo Acid Plant	1	7-20-76	5.2 (0.036)	5.3 (0.037)	5.6 (0.039)	10.7	11.0	11.0	99.8	31.9	2.1 (1,270)	0.4	1.1 (5.3)	2.3
	2	7-21-76	2.7 (0.019)	2.7 (0.019)	3.4 (0.024)	5.4	5.5	6.9	100.8	32.5	3.5 (2,110)	0.7	0.2 (1.0)	0.3
	3	7-22-76	1.6 (0.011)	1.7 (0.012)	2.5 (0.018)	3.4	3.5	5.2	99.5	27.1	3.7 (2,230)	0.8	1.1 (5.3)	2.3
	4	7-23-76	4.5 (0.032)	4.5 (0.032)	6.1 (0.043)	9.2	9.2	12.6	100.5	30.0	3.2 (1,930)	0.5	1.1 (5.3)	2.3
	5	7-26-76	3.4 (0.024)	3.4 (0.024)	4.2 (0.029)	6.0	6.1	7.5	97.6	26.1	4.1 (2,480)	0.7	104.8 (893)	331.0
	6	7-26-76	2.4 (0.017)	2.5 (0.018)	3.0 (0.021)	5.5	5.6	6.9	106.5	28.7	4.3 (2,600)	1.0	0.2 (1.0)	0.4
	7	7-27-76	10.5 (0.074)	10.6 (0.074)	11.3 (0.079)	22.6	22.7	24.3	97.5	30.5	4.6 (2,780)	1.0	0.2 (1.0)	0.5
	8	7-28-76	5.8 (0.041)	5.8 (0.041)	6.0 (0.042)	12.3	12.4	12.7	93.5	30.6	4.3 (2,600)	0.9	0.6 (2.9)	1.4
	9	7-28-76	3.9 (0.027)	3.9 (0.027)	5.2 (0.036)	8.0	8.9	10.8	98.1	30.6	2.6 (1,570)	0.5	1.0 (4.8)	2.1

[a] gr/scf

[b] ppm

T-808

TABLE 8-3. SUMMARY OF SAMPLING TIMES

Location	Run #	Date	Sampling Time		
Ajo Mainstack	1	7-20-76	A-Port	4:17 pm	4:37 pm
			B-Port	Not Running	
			C-Port	5:34 pm	5:54 pm
Ajo Reverb Esp	1	7-20-76	A-Port	4:15 pm	4:45 pm
			B-Port	4:58 pm	5:28 pm
Ajo Acid Plant	1	7-20-76	V-Port	4:15 pm	4:45 pm
			H-Port	5:20 pm	5:50 pm
Ajo Mainstack	2	7-21-76	A-Port	7:10 pm	7:30 pm
			B-Port	5:20 pm	5:40 pm
			C-Port	6:12 pm	6:32 pm
Ajo Reverb Esp	2	7-21-76	A-Port	5:25 pm	5:55 pm
			B-Port	6:00 pm	6:30 pm
Ajo Acid Plant	2	7-21-76	V-Port	5:22 pm	5:52 pm
			H-Port	6:24 pm	6:54 pm
Ajo Mainstack	3	7-22-76	A-Port	9:55 am	10:15 am
			B-Port	10:29 am	10:49 am
			C-Port	11:00 am	11:20 am
Ajo Reverb Esp	3	7-22-76	A-Port	9:56 am	10:26 am
			B-Port	10:29 am	10:59 am
Ajo Acid Plant	3	7-22-76	V-Port	10:02 am	10:32 am
			H-Port	11:10 am	11:40 am
Ajo Mainstack	4	7-23-76	A-Port	9:07 am	9:27 am
			B-Port	9:40 am	10:13 am
			C-Port	10:23 am	10:43 am
Ajo Reverb Esp	4	7-23-76	A-Port	9:18 am	9:48 am
			B-Port	9:53 am	10:23 am
Ajo Acid Plant	4	7-23-76	V-Port	10:12 am	10:42 am
			H-Port	9:10 am	9:40 am
Ajo Mainstack	5	7-26-76	A-Port	9:57 am	10:17 am
			B-Port	9:24 am	9:44 am
			C-Port	8:52 am	9:12 am
Ajo Reverb Esp	5	7-26-76	A-Port	8:50 am	9:20 am
			B-Port	9:25 am	9:55 am
Ajo Acid Plant	5	7-26-76	V-Port	8:55 am	9:25 am
			H-Port	10:10 am	10:40 am
Ajo Mainstack	6	7-26-76	A-Port	2:56 pm	3:16 pm
			B-Port	3:27 pm	3:47 pm
			C-Port	3:58 pm	4:18 pm
Ajo Reverb Esp	6	7-26-76	A-Port	2:56 pm	3:26 pm
			B-Port	3:27 pm	3:57 pm
Ajo Acid Plant	6	7-26-76	V-Port	4:00 pm	4:30 pm
			H-Port	2:55 pm	3:35 pm

T-27

T-27

TABLE 8-3. Concluded

Location	Run #	Date	Sampling Time		
Ajo Mainstack	7	7-27-76	A-Port	9:27 am	9:47 am
			B-Port	8:45 am	9:05 am
			C-Port	8:05 am	8:25 am
Ajo Reverb Esp	7	7-27-76	A-Port	8:35 am	9:05 am
			B-Port	9:08 am	9:38 am
Ajo Acid Plant	7	7-27-76	V-Port	9:45 am	10:15 am
				8:36 am	9:06 am
Ajo Mainstack	8	7-28-76	A-Port	11:16 am	11:36 am
			B-Port	11:55 am	12:15 pm
			C-Port	12:31 pm	12:51 pm
Ajo Reverb Esp	8	7-28-76	A-Port	11:13 am	11:43 am
			B-Port	11:44 am	12:14 pm
Ajo Acid Plant	8	7-28-76	V-Port	11:39 am	12:09 pm
			H-Port	12:22 pm	12:52 pm
Ajo Mainstack	9	7-28-76	A-Port	8:41 pm	9:01 pm
			B-Port	7:59 pm	8:19 pm
			C-Port	7:20 pm	7:40 pm
Ajo Reverb Esp	9	7-28-76	A-Port	7:16 pm	7:46 pm
			B-Port	7:49 pm	8:19 pm
Ajo Acid Plant	9	7-28-76	V-Port	8:23 pm	8:53 pm
			H-Port	7:19 pm	7:49 pm
Ajo Mainstack	10	7-29-76	A-Port	10:20 am	10:41 am
			B-Port	11:10 am	11:30 am
			C-Port	11:50 am	12:10 pm
Ajo Reverb Esp	10	7-29-76	A-Port	10:12 am	10:42 am
			B-Port	10:50 am	11:20 am
Ajo Mainstack	11	7-29-76	A-Port	5:15 pm	5:35 pm
			B-Port	4:37 pm	4:57 pm
			C-Port	4:02 pm	4:22 pm
Ajo Reverb Esp	11	7-29-76	A-Port	4:00 pm	4:30 pm
			B-Port	4:34 pm	5:04 pm
Ajo Mainstack	12	7-30-76	A-Port	11:15 am	11:35 am
			B-Port	11:58 am	12:18 pm
			C-Port	12:45 pm	1:05 pm
Ajo Reverb Esp	12	7-30-76	A-Port	11:20 am	11:50 am
			B-Port	11:55 am	12:25 pm

The sample recovery data shows that the residues obtained from the sampling nozzle, probe, and filter holder for Tests #1, #7, and #8 may be too high. The average residue weight for Tests #2, #4, #5, #6, and #9 was 47.03 mg. For Tests #1, #7, and #8, the residue weights were 98.55 mg, 244.80 mg, and 115.45 mg. It is possible that these higher-than-average residue weights are due to interaction of the sampling nozzle with the walls of the abnormally long sampling ports at the acid plant test site. For Test #3, the residue weight was only 9.16 mg. This residue weight was lower than the average because the glass liner in the sampling probe was broken during sample recovery and only a portion of the particulates was recovered.

A review of the field data sheets also shows a possible discrepancy in the velocity for Test #5. The average velocity (46.6 fps) for Test #5 was approximately 14 percent lower than the average velocity for all nine tests at the acid plant. This low velocity was noticed by the acid plant test crew while sampling on the horizontal traverse. Although the S-type pitot tube tips and connections were immediately inspected and cleaned, there was no change in the measured ΔP. However, an examination of the weight of material processed in the converter for Test #5 showed that it was the lowest amount for all nine tests. Hence, the decrease in velocity at the acid plant outlet was probably caused by a decrease in the amount of gas treated by the acid plant.

Finally, the particulate matter collected on the 142 mm filters was extremely hygroscopic in nature. Filter weights changed by as much as 17 to 72 percent because of the adsorption of water vapor from the ambient air during the weighing procedures. According to the daily activities report, most of the filters were only slightly soiled. Although no chemical analysis was performed on the filters, it seems likely that sulfuric acid mist was the main constituent of the matter collected.

Particulate Matter

A statistical analysis (see example calculation in Volume III, Appendix C) of the particulate emission rate data for the acid plant was carried out. In making this analysis the average residue weight (47.03 mg) was used for Tests #1, #7, and #8, since the measured values appeared to be too high. This same average residue weight was also used for Test #3, since the measured value seemed too low. Thus statistical analysis gave the following results:

Mean emission rate = 6.6 lb/hr

Standard deviation = 1.2

Based on these figures, there is 90-percent confidence that the measured particulate emission rate at the outlet of the acid plant is between ~~5.8~~ 5.9 lb/hr and ~~7.4~~ 7.3 lb/hr. This analysis was done using only the extrapolated weight data for the filters obtained from the weight versus time curves. This corrected data for the acid plant is summarized below:

Test	Emission Rate (lb/hr)
1	6.5
3	6.4
7	6.2
8	6.4

SO_3/H_2SO_4 (not including Test #5)

A statistical analysis of the SO_3/H_2SO_4 emission rate data yielded the following results:

Mean emission rate = 1.5 lb/hr

Standard deviation = 0.92

Based on this data, there is 90-percent confidence that the measured SO_3/H_2SO_4 emission rate is between 0.9 lb/hr and 2.1 lb/hr. Test #5 was omitted from the statistical analysis because it is an outlier. The analytical data and the computations of concentration and emission rate for this test were reviewed, but no errors were found. The sample was retitrated, but the result was practically the same as before. Apparently, the sample for Test #5 was contaminated with extraneous material in some unknown manner. Since no sample recovery took place in the field, it seems unlikely that foreign matter got into the IPA impingers during impinger train disassembly and transport of the impinger train to the laboratory. Since the laboratory itself was clean, contamination of the samples at this point was not a strong possibility either. One possible explanation is that the impinger train may have been improperly purged to remove any SO_2 trapped in solution. This could produce high SO_3/H_2SO_4 results. Although no specific reason could be found for the high result, Test #5 was rejected as not being representative.

SO_2

A statistical analysis of the SO_2 emission rate data gave the following results:

Mean emission rate = 0.7×10^3 lb/hr

Standard deviation = 0.2×10^3

On the basis of these data, there is 90-percent confidence that the measured SO_2 emission rate for the acid plant outlet is between 0.6×10^3 lb/hr and 0.8×10^3 lb/hr.

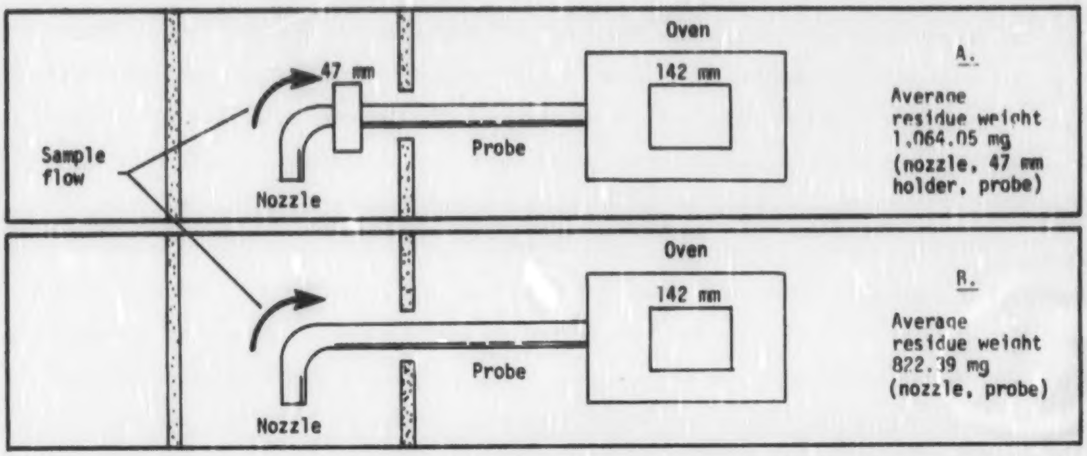
8.3.2 Reverb Furnace ESP Outlet Test Results

As was mentioned previously in the description of sample recovery and analysis, the glass fiber filters used in the sampling trains at the reverb furnace were difficult to remove from their stainless steel filter holders. For some unknown reason, the teflon coating on the 142 mm filter holder was attacked (probably chemically) during each test at this location, causing the teflon to break down and flake. Once portions of the teflon coating had flaked off the stainless steel, the glass fiber filters stuck to the stainless steel surfaces. As a result, when the filter was removed from the holder during sample recovery, visible amounts of glass fiber were left on the holder. Some of this filter material was removed, but in order to avoid contaminating the acetone washings with flakes of teflon, the filter holders were not washed, which means that any particulate matter and fibers adhering to the filter holder was not recovered. Hence, the reported filter weights and acetone residue weights are lower than the true weights. The size of this error cannot be determined.

Since the in-stack filter holders were also made of stainless steel, the 47 mm filters stuck to these surfaces as well as to the viton gasket and the stainless steel support screen. Every effort was made to recover as many of these fibers as possible. Since contamination with teflon was not a problem with these filter holders, the fibers and any particulates deposited on the internal surfaces of the holder could be recovered much more effectively than in the case of the 142 mm filter holders.

When the residue weights from the acetone washings of the nozzle and probe for Test #5 were compared to the weights for other tests, they were found to be lower than usual. The daily activities report indicated that the glass liner in the sampling probe was broken so that only a portion of the particulates were recovered.

The amount of particulate collected with the sampling train which used both an in-stack (47 mm) filter and an out-of-stack (142 mm) filter was consistently higher than the amount of particulate collected with the sampling train which used only an out-of-stack (142 mm) filter. The average residue weights obtained by washing the probe, nozzle, and 47 mm filter holder with acetone are (Test #5 was not included because of a broken glass liner in the sampling probe) shown on the following page:


Oven
A.
47 mm
142 mm
Average residue weight 1,064.05 mg (nozzle, 47 mm holder, probe)
Sample flow
Probe
Nozzle
Oven
B.
142 mm
Average residue weight 822.39 mg (nozzle, probe)
Probe
Nozzle

It was originally thought that this difference in weight was due to the fact that the in-stack (47 mm) filter holders could be rinsed with acetone whereas the out-of-stack (142 mm) filter holders could not (for fear of contamination with teflon flakes). If this were the case, the difference in the average residue weight for the two sampling trains would be accounted for by the weight of the unwashed residue on the 142 mm filter holder.

This seemed like a logical assumption, since in Sampling Train A above, most of the large particulate matter should have been removed in the nozzle, the 47 mm filter and the 47 mm filter holder and the probe before reaching the 142 mm filter holder — so that relatively little particulate would be lost in not washing the 142 mm filter holder. By contrast, in Sampling Train B above, it seemed that much more particulate should have been deposited on the 142 mm filter holder and lost when the holder could not be washed. In fact, however, over 1,000 mg of particulate were caught on the 142 mm filter in Sampling Train A — which means that much more fine particulate (including condensables at 250°F) was deposited on the 142 mm filter holder in Sampling Train A than was originally postulated. As a result, we have no clear explanation of the difference in the weight of the residues from the two sampling trains.

Test #7 was not included in the test results. Because the test crew failed to reconnect the tygon hose to the magnehelic gage after measuring static pressure, all velocity measurements and isokinetic sampling rates were in error. On top of this, a smelter electrical failure toward the end of the test caused a backup of the solutions in the impinger train. The test was a complete failure and was discarded.

All tests except #1 and #11 (in-stack test) were performed isokinetically. Tests #1 and #11 were conducted under subisokinetic conditions, which means that the measured particulate emission rates are erroneously high. However, since the tests were conducted downstream of the electrostatic precipitator where most of the particles are less than 5μ in size, the effect of the nonisokinetic conditions on the emission rates is probably small. This supposition is supported by the emission data in Table 8-2. The emission rates for Tests #1 and #11 are not drastically different from the rates for the other tests. Nevertheless, these results should be rejected because the isokinetic guideline was not met.

The average velocity for Test #5 was lower than average velocities measured during all other tests at this reverb furnace test site. Since the ΔP data for the Method 3 and 4 train compare favorably with the data for the Method 5 and 8 train, the decrease in velocity appears to have been caused by process changes. Both trains were sampling at the same time and both S-type pitot tubes produced similar data. Since it is unlikely that both pitot tubes were measuring velocity incorrectly, the feed rate of concentrate to the reverb furnace probably decreased during a portion of this particular stack test.

The highest average velocity was measured during Test #8. This test was conducted with the fugitive gas collection system off. Test #9 was also conducted with the fugitive gas collection system off, but the velocity at the test station was approximately 8 percent lower. The reason for this difference is not immediately clear, since the fugitive gas collection system operates with its own fans and is independent of the ID fans on the precipitator. The difference seems to have been caused by changes in process conditions, and no correction of the velocity data appears to be warranted.

The particulate matter caught on all the filters used in reverb furnace ESP outlet tests had hygroscopic characteristics. The differences between the extrapolated weights and the equilibrium weights for the 142 mm filters varied from approximately 30 to 50 percent. Even the particulates on the in-stack 47 mm filters were hygroscopic in nature, with weight gains varying from approximately 20 to 40 percent above the extrapolated weight values. Again, no chemical analysis of the filter samples was conducted, but it seems likely that a good portion of the particulate collected was sulfuric acid mist.

Table 8-4 compares particulate emission rate data from in-stack sampling tests with data from the out-of-stack or EPA Method 5 tests. In figuring the in-stack emission rate, residue from the nozzle, 47 mm filter holder, and probe were included with the in-stack filter weight. It is quite obvious that a great deal of particulate matter passes right through the in-stack filter. This

T-270

TABLE 8-4. COMPARISON OF EPA METHOD 5 AND IN-STACK/OUT-OF-STACK SAMPLING TECHNIQUES

Location	Run No.	EPA Method 5 Extrapolated Weight (lb/hr)	In-Stack Extrapolated Weight (lb/hr)	In-Stack/Out-of-Stack Extrapolated Weight (lb/hr)
Main Stack	1	r	nd	nd
	2	553.3	nd	nd
	3	r	nd	nd
	4	r	nd	nd
	5	458.7	nd	nd
	6	504.7	nd	nd
	7	r	nd	nd
	8	r	nd	nd
	9	r	nd	nd
	Average	505.6	--	--
	10	nd	489.9	520.0
	11	nd	500.7	513.9
	12	nd	573.2	588.9
	Average	--	524.3	540.9
Reverb ESP	[illegible]	r	nd	nd
	2	318.2	nd	nd
	3	272.9	nd	nd
	4	216.2	nd	nd
	5	255.9[a]	nd	nd
	6	330.3	nd	nd
	7	r	nd	nd
	8	331.3	nd	nd
	9	273.3	nd	nd
	Average	285.4	--	--
	10	nd	217.2	423.0
	11	nd	r	r
	12	nd	208.4	423.9
	Average	--	212.8	423.5

[a] Corrected emission data

particulate matter may be submicron in nature (<0.3μ) or may be gaseous at the stack temperature (e.g., SO_3).

It is clear from these data that using an in-stack sampling technique such as in-stack 47 mm glass filter on a hot source such as the reverb furnace is not equivalent to EPA Method 5.

Particulate Matter

A statistical analysis of the particulate emission rate data was made based on the following assumptions:

- Tests #1, #7, and #11 were rejected
- Measured emission rate data are erroneously low due to the inability to recover particulates from the 142 mm filter holders
- Emission rates are measured by two different methods — in-stack and out-of-stack
- An average residue weight (872.39), based on the out-of-stack residue data only, was used in place of the measured value for Test #5
- The corrected emission rate for Test #5 is 255.9 lb/hr

This analysis gave the following results:

- Mean emission rate = 316.1 lb/hr
- Standard deviation = 71.4

Based on these results, there is a 90-percent confidence that the measured particulate emission rate is between 271.8 lb/hr and 360.4 lb/hr.

An examination of the SO_3/H_2SO_4 emission data in Table 8-2 shows that on two occasions (Tests #5 and #8) no SO_3/H_2SO_4 was trapped in the IPA impingers. Since this occurrence is unlikely, the field samples were reanalyzed; the results were the same, however -- no SO_3/H_2SO_4 was found. Reviewing the daily activities report showed that the first IPA impinger for Test #5 was a ginger-ale color instead of being clear. Also, during the purge of the sampling train after the test, a crack was noted in the inlet to the 50 mm filter holder. While a crack in this location should not affect the outcome of the test for SO_3/H_2SO_4 (since the filter is after the IPA impingers), the discoloration of the impinger solution suggests the presence of some unknown element which may have interferred with the titration. No attempt was made to identify this element.

For Test #8, although no discoloration of the IPA impinger solutions was observed, the 50 mm filter following the IPA impingers had a light yellow color which looked like elemental sulfur. It is not clear what type of reducing agent would be present in the reverb gas stream to convert SO_3/H_2SO_4 and SO_2 to elemental S. Because of these unexplained anomalies, results of Tests #5 and #8 for the reverb furnace were rejected.

As mentioned previously, there were no results for Test #7 since a power failure during the test caused a sharp loss in pump vacuum which in turn caused the impinger solutions to back up and ruin the sample.

SO_3/H_2SO_4

A statistical analysis of the SO_3/H_2SO_4 emission rate data (excluding Tests #5, #7, and #8) gave the following results:

Mean emission rate = 24.9 lb/hr

Standard deviation = 12.4

Based on this data, there is 90-percent confidence that the measured emission rate is between 17.2 lb/hr and 32.6 lb/hr.

An examination of the SO_2 emission rate data in Table 8-2 shows that the results from Tests #5, #9, and #11 are lower than the results of other tests. Test #5 was retitrated, but the same results were obtained. One possible reason for the low results from Test #5 may be the crack found in the 50 mm filter holder inlet after the test was completed. It is not known when this crack occurred. This crack may have happened during disassembly of the impinger train after completion of the test, since the sampling train passed the leak check before the start of B traverse. But, if the crack occurred during the traverse rather than after it, the SO_2 concentration would be diluted because ambient air would have entered the system through the crack.

The reverb furnace log sheets were reviewed to determine whether there had been a significant change in the kind (concentrate, converter slag, precipitate, lime, dust) or the amount of material fed to the furnace which might account for the differences in the amount of SO_2 measured from one test to another. There did not appear to be any correlation of this kind. In fact, for Test #6, the percentage of all input materials as well as the total amount of feed to the furnace was lower than for any other test, yet the SO_2 emission rate was the highest measured. This can be explained by the fact that the gross weight of a particular component fed to the furnace is only one of a number of factors affecting SO_2 emissions. The percentage of sulfur in each component can vary considerably from day to day — in which case variations in the amount of SO_2 emitted would occur. Unfortunately, this program did not include taking samples of the material fed to the reverb furnace or analyzing them for sulfur. (Normally, the sulfur content of the concentrate fed to this type of furnace varies between 25 and 30 percent by weight.)

One other important factor is the manner in which the reverb furnace is fed. Normally, the concentrate is slung into the furnace every 15 minutes. Since the process observer had other processes to observe and was not permanently stationed at the reverb furnace, it is not possible to determine if the normal charging practices were strictly followed. Hence, any variation in the number of times the furnace was fed during a given test could also affect the amount of SO_2 emitted. To sum it all up, the variation in SO_2 concentration experienced at the reverb test site cannot be traced to a single process variable and appears to be the result of a combination of factors.

SO_2

A statistical analysis of the SO_2 emission rate data (excluding Test #7) gave the following results:

Mean emission rate = 5.1 x 10^3 lb/hr

Standard deviation = 2.5 x 10^3

Based on the above data, there is 90-percent confidence that the measured SO_2 emission rate is between ~~3.6~~ 3.7 x 10^3 lb/hr and ~~6.6~~ 6.5 x 10^3 lb/hr.

8.3.3 Main Stack Test Results

The analysis of these test results are split into two sections – test results with the fugitive gas collection system on and test results with the fugitive gas collection system off (Tests #8 and #9).

8.3.3.1 Fugitive Gas Collection System On

Tests #1 to #7, and #10 to #12 were conducted with the fugitive gas collection system on. This system collects fugitive emissions from the reverb furnace and the converter and transports these emissions directly to the main stack in separate ducts. Neither of these gas streams have particulate or gaseous pollutant removal devices. In addition to these two fugitive emission streams, the effluent from the acid plant and reverb furnace ESP also enter the main stack.

A review of the field data sheets showed that Test #1 on the main stack was conducted poorly. The crew experienced a great deal of difficulty with the sampling equipment (leaks in various components) and the movement from port to port. Because of these difficulties, Test #1 was conducted with a leak rate of 0.1 cfm at 15 inches Hg and included only Traverses A and C (but not B). As a result Test #1 is not considered a valid test result.

The major leak in the sampling equipment for the main stack was traced to the teflon hose used to connect the 142 mm filter to the impinger train. Once the fittings on the hose were repaired, no further difficulties were encountered. The data sheets for Test #2 showed no problems during the test, except that the probe and oven temperatures were slightly less than 250°F.

During A-traverse of Test #3, the stack gas velocity rose substantially for the first six traverse points then dropped back to normal for the remainder of the traverse and the test. There is no explanation for this momentary increase in velocity, which forced the vacuum pump wide open and made it difficult for the crew to maintain isokinetic sampling conditions. As a result, the percent isokinetic sampling was 87.8, which is less than the minimum acceptable value of 90. This means that the measured particulate emission rate is probably a little higher than its actual value, since the sampling was conducted at subisokinetic conditions. Because of this, the results of this test should be rejected.

During Test #4, the roof monitor began to smoke. The problem was traced to closed dampers in the Cottrell precipitator, which meant that little or no suction was being applied to the fugitive gas collection system. This problem was corrected before the second traverse began so that only the first traverse was affected. Test #4 was also conducted under subisokinetic sampling conditions and hence, the measured mass emission rate is higher than its actual value. This result should also be rejected for failing to satisfy the isokinetic sampling criteria.

Tests #5 and #6 were conducted isokinetically, but the average velocity in the stack was lower than for the previous tests. This decrease in velocity was probably caused by a decrease in the amount of material processed in the converter and the reverb furnace.

A breakdown in communications prior to the start of Test #7 caused the test crew to complete Traverse C before copper blow had started and before the other crews had started sampling. As a result of this early start, the average stack gas velocity was lower than usual. This low velocity was probably caused by the acid plant operation. Just before copper blow occurs in the converter, several operations (skimming, addition of chips, etc.) take place at the converter. During these operations, the acid plant is not operating, so that no gas is flowing to the main stack from the acid plant. The acid plant comes on-line as soon as copper blow is started. The fact that the acid plant was not operating caused the measured mass emission rate for Test #7 (333.7 lb/hr) to be low because the velocity and volumetric stack gas flowrate were lower than for the other tests. In addition, the isokinetic sampling criteria was exceeded for this test (115.2 percent). Superisokinetic sampling conditions also produce artificially low results.

Tests #10, #11, and #12 were all conducted with the in-stack 47 mm filters. Although all tests were done isokinetically, one minor problem did occur with particulate sample recovery. As discussed previously in Section 8.3.2, the glass fiber filters stuck to the support screen and gasket in the 47 mm filter holder. Recovery of the stuck fibers was nearly quantitative, but some fibers were probably left behind. Likewise, the same comments can be made about in-stack versus out-of-stack sampling for the main stack as were made previously for the reverb furnace tests in Section 8.3.2 – namely that the in-stack filter method is not equivalent to EPA Method 5. For the main stack tests, approximately 12.6 lb/hr to 21.7 lb/hr – or about 2.4 to 4.1 percent of the total particulate emission rate – were not measured by the in-stack filter. These values are an order of magnitude lower than the difference between the in-stack and out-of-stack methods at the reverb ESP outlet because the main stack gas temperature was so much lower than the reverb ESP gas temperature.

A summary of the valid particulate matter emission rate data is presented in Table 8-5. As indicated previously, the sum of the emissions from the acid plant, the reverb ESP and the fugitive gas collection system should equal the emissions measured at the main stack (see Figure 8-3). Also, the difference between the emission rate at the main stack and the sum of the emission rates at the acid plant and reverb ESP should equal the emissions rate of the fugitive gas collection system. The table shows that the fugitive gas collection system contributes a substantial amount of particulate matter. These emission rates will be discussed in more detail in the next subsection.

8.3.3.2 Fugitive Gas Collection System Off

Tests #8 and #9 were conducted with the fugitive gas collection system off. It was hoped that the decrease in the mass emission for the main stack would be a direct result of turning the fugitive gas collection system off. These average mass emission rates could then be compared with those mass emission rates measured at the main stack with the fugitive gas collection system on to get an estimate of the particulate contribution made by the fugitive gas collection system. At the main stack, Tests #8 and #9 were conducted at superisokinetic sampling conditions, which means the measured particulate emission rates are underestimated. Even taking into account these superisokinetic conditions, the amount of particulate matter on the filter for Test #8 is very small compared to the other filter catches. Since there is no mention of a problem recovering the filter from the holder on the daily activities report (i.e., low filter weight not due to lost fibers), there is no explanation for this low result. This data was analyzed to check whether or not the emission rate of the main stack was approximately equal to the sum of the emission rates of the acid plant and the reverb furnace (fugitive system off). This analysis gave the following results: one negative

TABLE 8-5. VALID PARTICULATE EMISSION RATE DATA

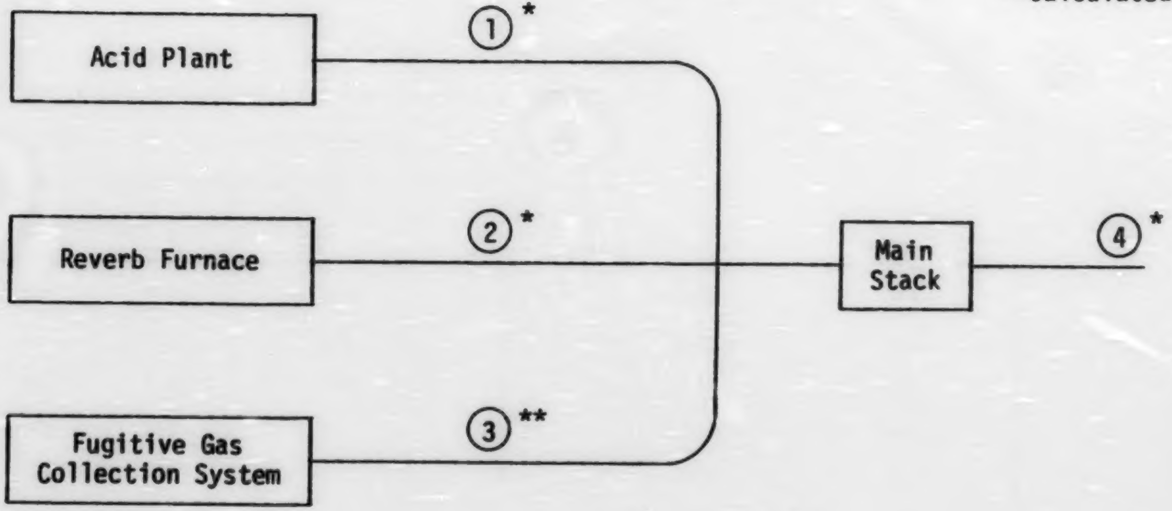
Run #	Acid Plant (lb/hr)	Reverb ESP (lb/hr)	Acid Plant + Rev. ESP (lb/hr)	Main Stack (lb/hr)	Fugitive Gas Collection System (lb/hr)
1	6.5[b]	r	--	r	--
2	5.4	318.2	323.6	553.3	229.7
3	6.4[b]	272.9	279.3	r	--
4	9.2	216.2	225.4	r	--
5	6.0	255.9[b]	261.9	458.7	196.8
6	5.5	330.3	335.8	504.7	168.9
7	6.2[b]	r	--	r	--
8[c]	6.4[b]	331.3	337.7	r	--
9[c]	8.0	273.3	281.3	r	--
10	6.6[a]	423.0	429.7	520.0	90.3
11	6.6[a]	r	--	513.9	--
12	6.6[a]	423.9	430.6	588.9	158.3

r = Rejected

[a] Not sampled, average value of Tests 1 through 9

[b] Corrected emission data (used average residue weight of 47.03 mg in place of measured weights for Tests 1, 3, 7, 8 at acid plant; used average residue weight of 872.39 mg in place of measured weight for Test 5 at reverb.

[c] Fugitive system off


Acid Plant
Reverb Furnace
Fugitive Gas Collection System
Main Stack
① *
② *
③ **
④ *

*Measured value
**Calculated by difference

Case A: Fugitive Gas Collection System On (Tests #1 through #7, #10 through #12)

$$\textcircled{4}_{A} - (\textcircled{1} + \textcircled{2}) = \textcircled{3}$$

Case B: Fugitive Gas Collection System Off (Tests #8 and #9)

$$\textcircled{4}_{B} = \textcircled{1} + \textcircled{2}$$

Figure 8-3. Difference in measurements calculation.

TABLE 8-6. GAS FLOW CONTRIBUTION OF FUGITIVE GAS COLLECTION SYSTEM

T-83

Run #	Acid Plant (scfh x 10^6)	Reverb ESP (scfh x 10^6)	Acid Plant + Reverb ESP (scfh x 10^6)	Main Stack (scfh x 10^6)	Fugitive Gas Collection System (scfh x 10^6)
1	2.1	3.1	5.2	14.0	8.8
2	2.0	3.6	5.6	14.0	8.4
3	2.9	3.0	5.9	18.5	12.6
4	2.0	3.8	5.8	15.0	9.2
5	1.8	2.8	4.6	11.9	7.3
6	2.3	3.6	5.9	10.8	4.9
7	2.1	r	--	7.8	--
8[b]	2.1	4.2	6.3	8.0	1.7
9[b]	2.1	3.8	5.9	11.3	5.4
10	2.1[a]	3.9	6.0	11.8	5.8
11	2.1[a]	3.9	6.0	10.0	4.0
12	2.1[a]	3.6	5.7	13.6	7.9

r = Rejected

[a] Not measured, average values

[b] Fugitive gas collection system off

[c] Difference between main stack and (reverb + acid plant)

emission rate (Test #8, -167.7 lb/hr) which is impossible and one positive emission rate (Test #9, 566.4 lb/hr). The proposed comparison of emission rates could not be made because of the contradictory results which were obtained.

Hence, it was decided to use the difference in methods calculation (Case A, Figure 8-3) to determine the contribution of particulate matter by the fugitive gas collection system. Average particulate emission rate data (Table 8-5) and average volumetric flowrate data (Table 8-6) calculated for the fugitive gas collection system were as follows:

$$\left.\begin{array}{l}\text{Average volumetric gas flowrate} = 7.7 \times 10^6 \text{ scfh}\\ \\ \text{Average particulate emission rate} = 168.8 \text{ lb/hr}\end{array}\right\}\text{ Tests \#2, \#5, \#6, \#10, and \#12}$$

As a check of this data, the rated capacities for the fans in the fugitive gas collection system were obtained from Phelps-Dodge as indicated in Table 8-7. The total rated capacity of the system is approximately 8.4 x 10^6 scfh. Since these fans are old (installed approximately 1960) and have been modified over the years, they do not run at rated capacity. Their actual capacity is somewhat lower, probably very close to the average value of 7.7 x 10^6 scfh determined during the test program.

Particulate Matter

A statistical analysis of the particulate emission rate data using data from Tests #2, #5, #6, #10, #11, and #12 yielded the following results:

Mean emission rate = 523.3 lb/hr

Standard deviation = 44.3

Based on this data, there is 90-percent confidence that the measured particulate emission rate is between 486.9 ~~304.6~~ lb/hr and 559.7 ~~741.9~~ lb/hr.

As can be seen from the data in Tables 8-8 and 8-9, the interpretation of the SO_3/H_2SO_4 and SO_2 emission rate data is somewhat more difficult. Since the SO_3/H_2SO_4 and SO_2 caught in the impinger train are in the gaseous state, the measured concentrations are not subject to errors associated with isokinetic sampling. This means that much more of the test data are available for review. Since the results from Tests #2 and #8 seemed low and the results from Test #6 appeared high

TABLE 8-7. RATED FAN CAPACITIES IN FUGITIVE GAS COLLECTION SYSTEM[a]

Converter — 70,000 acfm at 350°F and 28 inches Hg
(42,863 scfm at 70°F and 29.92 inches Hg)

Reverb Matte Tunnel — 60,000 acfm at 300°F and 28 inches Hg
(39,157 scfm at 70°F and 29.92 inches Hg)

Matte Launder — 60,000 acfm at 300°F and 28 inches Hg
(39,157 scfm at 70°F and 29.92 inches Hg)

Slag Launder — 30,000 acfm at 300°F and 28 inches Hg
(19,579 scfm at 70°F and 29.92 inches Hg)

Total = 140,756 scfm or 8.4×10^6 scfh

[a] Best available information from Phelps-Dodge

TABLE 8-8. VALID SO_3/H_2SO_4 EMISSION RATE DATA

T-84

Run #	Acid Plant (lb/hr)	Reverb (lb/hr)	Acid Plant + Reverb (lb/hr)	Main Stack (lb/hr)	Fugitive Gas Collection System (lb/hr)
1	2.3	10.5	12.8	r	--
2	0.3	37.2	37.5	1.6	-35.9
3	2.3	16.8	19.1	6.4	-12.7
4	2.3	2.3	4.6	22.4	+17.8
5	r	r	--	10.8	--
6	0.4	24.0	24.4	94.1	+69.7
7	0.5	r	--	r	--
8[b]	1.4	r	--	r	--
9[b]	2.1	33.7	35.8	8.5	-27.3
10	1.5[a]	33.0	34.5	16.8	-17.7
11	1.5[a]	32.7	34.2	8.1	-26.1
12	1.5[a]	33.9	35.4	6.3	-29.1

r = Rejected

[a] Not sampled, average data

[b] Fugitive system off

TABLE 8-9. VALID SO_2 DATA

T-85

Run #	Acid Plant (lb/hr)	Reverb (lb/hr)	Acid Plant + Reverb (lb/hr)	Main Stack (lb/hr)	Fugitive Gas Collection System (lb/hr)
1	400	5,000	5,400	r	--
2	700	9,500	10,200	6,400	- 3,800
3	800	3,300	4,100	8,200	+ 4,100
4	500	4,000	4,500	6,300	+ 1,800
5	700	2,200	2,900	8,400	+ 5,500
6	1,000	9,500	10,500	100	-10,400
7	1,000	r	--	r	--
8[b]	900	3,800	4,700	3,000	- 1,700
9[b]	500	1,100	1,600	700	- 900
10	722[a]	4,800	5,522	5,900	+ 378
11	722[a]	2,400	3,122	3,900	+ 778
12	722[a]	5,000	5,722	5,500	- 222

r = Rejected

[a] Not sampled, average value

[b] Fugitive system off

in comparison to the other test results, these samples were retitrated to determine if an analytical error had been made. No such error was discovered. Likewise, the emission rate calculations were rechecked and no error was found. The daily activities report noted no problems with sample recovery for Tests #2 and #8. There was a light yellow color on the 50 mm filter in back of the IPA impingers which were clear. Neither the composition of this yellow substance (looks like elemental sulfur) nor its effect on the titration results are known. Also, for Test #6 the only deviation from routine procedures noted was the fact that the 50 mm filter holder was reversed (the gas was filtered through the frit and then the filter instead of the other way around). High SO_3/H_2SO_4 results for Test #6 could have resulted from insufficient purging of the solutions to remove any SO_2 trapped in the IPA solutions, but a check of the field data sheet showed that the impinger train was purged.

Test #1 was rejected because the sampling train never passed the leak check, which caused the measured value to be low. Test #7 was rejected because the test was started before copper blow began.

In six of the eight tests, the amount of SO_3/H_2SO_4 measured at the main stack sampling site was less than the sum of the inputs from the acid plant, reverb furnace and fugitive gas collection system. This meant that the fugitive gas collection system was not contributing SO_3/H_2SO_4 emissions but was acting as a sink for SO_3/H_2SO_4 emissions — which is impossible. One explanation for this phenomenon is condensation - that is, some of the gaseous SO_3 measured at the acid plant and reverb furnace ESP outlets became sulfuric acid mist when the two streams mixed at the base of the main stack as the gas temperature dropped. This sulfuric acid mist would probably have been caught in the front half on the Method 5/8 sampling train on the main stack and would never have reached the IPA impingers. If this condensation process did occur, higher particulate emissions were probably attributed to the fugitive gas collection system (difference between main stack measurement and sum of acid plant and reverb furnace measurements) than this system actually contributed, since a good portion of these emissions seems to be attributable to this condensation phenomenon which occurs when the acid plant and reverb furnace gas streams mix.

The two positive SO_3/H_2SO_4 values measured at the main stack are probably the result of process variability which will be discussed in more detail for the SO_2 results. As mentioned previously, no error was made in the analysis or computation, so that no explanation of these results can be suggested at this time.

SO_3/H_2SO_4

A statistical analysis of the SO_3/H_2SO_4 emission rate data (excluding Tests #1, #7, and #8) yielded the following results:

Mean emission rate = 19.4 lb/hr

Standard deviation = 28.7

On the basis of this data, there is 90-percent confidence that the SO_3/H_2SO_4 emission rate is between 1.6 lb/hr and 37.2 lb/hr.

It should be noted, however, that these results are somewhat misleading, because they are based on the concentration of SO_3/H_2SO_4 measured only in the IPA impingers and on the back-up 50 mm filter. Although no chemical analysis was performed on the Method 5 filters (142 mm filter at 250°F), it seems as if a significant amount of sulfuric acid mist was deposited on the filter since the material was very hygroscopic in nature. In this situation, what gets classified as particulate and what gets classified as SO_3/H_2SO_4 is dictated by the Method 5 definition of particulate matter — anything caught on a glass fiber filter at 250°F is particulate matter.

The SO_2 emission rate data for the main stack appeared to be consistent, but the calculated values for the fugitive gas collection system displayed erratic tendencies. Half of the tests in the main stack showed positive results (that is, a positive contribution by the fugitive gas collection system) while the other half of the data gave negative results. A review of the main stack test data showed only one result which appeared to be suspicious — Test #6. Once again, the field sample was retitrated and the emission rate calculation was rechecked, but no error could be found. There is one possible explanation for these erratic results: All tests were conducted during the copper blow, but not at exactly the same time. There are several reasons for this:

- The number of sampling points and hence the time required to conduct a traverse differed for each test location
- The number of traverses differed at the main stack
- The degree of difficulty for equipment transfer from port to port differed considerably

Because of these factors, it is quite possible that the test crew at the reverb test site was sampling during a time when the furnace was being charged with concentrate (which causes a spike of SO_2 emissions which taper off with time as the concentrate melts) while the main stack test crew were transfering their sampling equipment from one port to another. The result of a situation like this would be a high SO_2 measurement at the reverb test site and a low SO_2 measurement at the main stack. This in turn would produce negative (and seemingly impossible) results for the fugitive gas collection system.

Similarly, the crew at the reverb test site usually required less time to complete a 1-hour test than either of the other two crews because of the relative ease of transfering from one sample port to another (approximately 5 minutes). As a result, it is possible that the reverb furnace could have been fed after the reverb test crew had finished their test but before the main stack test crew had finished theirs. The result in this case would be a lower SO_2 emission rate at the reverb, a higher SO_2 emission rate at the main stack, and a positive result for the fugitive gas collection system. Phelps-Dodge does not keep a record of the time the reverb furnace is charged, so there is no means to verify the exact time the furnace was charged and whether or not the reverb test crew and the main stack test crew were sampling at this precise moment. Hence, the calculated SO_2 emission rate data for the fugitive gas collection system (difference in measurements between main stack and reverb furnace plus acid plant) is probably not representative of actual conditions.

SO_2

A statistical analysis of the main stack SO_2 emission rate data (excluding Tests #1, #6, and #7) yielded the following results:

Mean emission rate = 5.4×10^3 lb/hr

Standard deviation = 2.5×10^3

Based on this data there is 90-percent confidence that the measured SO_2 emission rate is between 3.85×10^3 lb/hr and 6.95×10^3 lb/hr.

8.4 CONCLUSIONS

The following conclusions were drawn after an in-depth review of the test results.

- The emissions of particulate matter from the reverb furnace ESP and the main stack at the Phelps-Dodge smelter in Ajo, Arizona are far in excess of the allowable particulate emission rates.
- Because of process variability, any future tests conducted at all three locations (acid plant, reverb, main stack) should be done exactly at the same time to eliminate emission variation as a function of time.
- The use of in-stack glass fiber filters to measure the emission rate of particulate matter in place of EPA Method 5 is an unacceptable procedure which yields erroneously low results.

- The values of the fugitive gas collection system particulate emission data and volumetric gas flowrate obtained by the difference in measurements calculation procedure appears to indicate a significant contribution to the atmosphere.
- The emission contribution of the fugitive gas collection system should be determined by direct measurement or by the difference in measurements calculation procedure only if the three locations (acid plant, reverb, main stack) are tested at precisely the same time.
- The particulate matter caught on the glass fiber filters maintained at 250°F is very hygroscopic in nature. The chemical analysis of this particulate matter should be undertaken to determine the nature of this material and to indicate the type of control equipment needed to effectively remove it.